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NEWS	3	FEB 25	CA/CAPLUS - Russian Agency for Patents and Trademarks (ROSPATENT) added to list of core patent offices covered
NEWS	4	FEB 28	PATDPAFULL - New display fields provide for legal status data from INPADOC
NEWS	5	FEB 28	BABS - Current-awareness alerts (SDIs) available
NEWS	6	FEB 28	MEDLINE/LMEDLINE reloaded
NEWS	7	MAR 02	GBFULL: New full-text patent database on STN
NEWS	8	MAR 03	REGISTRY/ZREGISTRY - Sequence annotations enhanced
NEWS	9	MAR 03	MEDLINE file segment of TOXCENTER reloaded
NEWS	10	MAR 22	KOREAPAT now updated monthly; patent information enhanced
NEWS	11	MAR 22	Original IDE display format returns to REGISTRY/ZREGISTRY
NEWS	12	MAR 22	PATDPASPC - New patent database available
NEWS	13	MAR 22	REGISTRY/ZREGISTRY enhanced with experimental property tags
NEWS	14	APR 04	EPFULL enhanced with additional patent information and new fields
NEWS	15	APR 04	EMBASE - Database reloaded and enhanced
NEWS	16	APR 18	New CAS Information Use Policies available online
NEWS	17	APR 25	Patent searching, including current-awareness alerts (SDIs), based on application date in CA/CAplus and USPATFULL/USPAT2 may be affected by a change in filing date for U.S. applications.
NEWS	18	APR 28	Improved searching of U.S. Patent Classifications for U.S. patent records in CA/CAplus

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=> s 1738-25-6/rn
           305 1738-25-6
             5 1738-25-6D
L1         302 1738-25-6/RN
                 (1738-25-6 (NOTL) 1738-25-6D )

=> s 109-55-7/rn
          3765 109-55-7
          1117 109-55-7D
L2        2730 109-55-7/RN
                 (109-55-7 (NOTL) 109-55-7D )

=> s l1 and l2
L3          44 L1 AND L2

=> d l3 1-44 abs ibib
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L3 ANSWER 1 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali is described.

ACCESSION NUMBER: 2004:609968 CAPLUS
DOCUMENT NUMBER: 141:140075
TITLE: Low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 8 pp., Cont.-in-part of U.S. Ser. No. 327,765.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004147784	A1	20040729	US 2003-731733	20031209
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A2 20021223
US 2003-731733 A 20031209
OTHER SOURCE(S): CASREACT 141:140075

L3 ANSWER 2 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the production of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile (I) comprises: feeding hydrogen and 3-(dimethylamino)propionitrile into a low-pressure reactor containing a sponge nickel catalyst, at least one Group IA alkali metal hydroxide (e.g., potassium hydroxide), and water to form a reaction medium; heating the reaction medium to 70-100°; pressurizing the reactor to 45-500 psig; and hydrogenating the nitrile to form I.

ACCESSION NUMBER: 2004:589527 CAPLUS
DOCUMENT NUMBER: 141:123405
TITLE: Low-pressure catalytic hydrogenation process for the manufacture of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 6660887	B1	20031209	US 2002-327765	20021223
US 2004147784	A1	20040729	US 2003-731733	20031209

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209
OTHER SOURCE(S): CASREACT 141:123405

L3 ANSWER 3 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB A process for the production of 3-(dimethylamino)propylamine (I) in high (>99%) purity from 3-(dimethylamino)propionitrile utilizing a low-pressure hydrogenation process is described which comprises contacting the nitrile with hydrogen at low pressure in the presence of a sponge nickel catalyst and ≥1 Goup IA metal hydroxide at 70-100°/45-150 psig. The improvement in the process resides in a combination of carrying out the hydrogenation process at low pressures and temps. in the presence of a catalytic amount of caustic base in order to give a I selectivity of >99.60%.

ACCESSION NUMBER: 2003:961180 CAPLUS
DOCUMENT NUMBER: 140:17730
TITLE: Low-pressure hydrogenation process and catalyst system for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060039	A2	20040722	WO 2003-US29721	20030919
WO 2004060039	A3	20040826		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004147784	A1	20040729	US 2003-731733	20031209
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209
OTHER SOURCE(S): CASREACT 140:17730
REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 4 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines were prepared by hydrogenation of nitriles in the presence of catalysts containing Co and optionally Ni as well as ≥1 doping metal on a particulate substrate, whereby the Co and optional Ni have an avg. particle size of 3-30 nM. Thus, dimethylaminopropionitrile was hydrogenated in the presence of a suspension catalyst [prepared from Co(NO3)2, Ni(NO3)2, and Y(NO3)3 and aluminosilicate powder] at 80° in the presence of NH3 and 80 bar H2 to give dimethylaminopropylamine in 98.4% selectivity.

ACCESSION NUMBER: 2003:332011 CAPLUS
DOCUMENT NUMBER: 138:337704
TITLE: Preparation of primary amines via reduction of nitriles in the presence of supported cobalt catalysts containing dopants and optionally containing nickel.
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 14 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10152135	A1	20030430	DE 2001-10152135	20011023
US 2003120115	A1	20030626	US 2002-271977	20021017
US 6790996	B2	20040914		
EP 1306365	A2	20030502	EP 2002-23640	20021021
EP 1306365	A3	20031015		

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK

JP 2003192647	A2	20030709	JP 2002-307884	20021023

PRIORITY APPLN. INFO.: DE 2001-10152135 A 20011023
OTHER SOURCE(S): CASREACT 138:337704; MARPAT 138:337704

L3 ANSWER 5 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Organic compds. are hydrogenated by bringing into contact with a H-containing gas in the presence of a catalyst comprising, as active metal, Ru alone or together with ≥1 group I, group VII or group VIII metal. The catalyst is prepared by (i) contacting amorphous SiO2 support with halogen-free aqueous solns. of a low-mol.-weight Ru compound, (ii) drying at 200° and (iii) hydrogenating at 100-350°. The step (ii) is accomplished directly following the step (i). For example, hydrogenation of 100 g diisononyl phthalate at 120° and 200 bar H in the presence of 2 g Ru/SiO2 catalyst (3% Ru on SiO2 powder; preparation given) gave 1,2-cyclohexanedicarboxylic acid diisononyl ester with 99% selectivity.

ACCESSION NUMBER: 2002:941599 CAPLUS
DOCUMENT NUMBER: 138:26087
TITLE: Procedure and ruthenium catalyst for hydrogenation of organic compounds
INVENTOR(S): Boettcher, Arnd; Vanoppen, Dominic; Arndt, Jan-Dirk; Henkelmann, Jochem
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 22 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10128242	A1	20021212	DE 2001-10128242	20010611
WO 2002100536	A1	20021219	WO 2002-EP6287	20020607
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1412082	A1	20040428	EP 2002-740699	20020607
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
US 2004199033	A1	20041007	US 2003-480196	20031210
PRIORITY APPLN. INFO.:			DE 2001-10128242	A 20010611
			WO 2002-EP6287	W 20020607

L3 ANSWER 6 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Mixts. of primary amines XCHZNH2 [e.g., 3-(dimethylamino)propylamine] and secondary amines (XCH2)2NH [X = (un)substituted alkyl, cycloalkyl, dialkylamino, etc.; e.g., bis[3-(dimethylamino)]propylamine] are obtained from nitriles XCN [e.g., 3-(dimethylamino)propionitrile] by hydrogenation of the nitrile at 50-250°/5-350 bars in the presence of a hydrogenation catalyst containing 0.1-10% palladium on a support (e.g., zirconia).

ACCESSION NUMBER: 2002:517942 CAPLUS
DOCUMENT NUMBER: 137:80624
TITLE: Hydrogenation process and palladium-containing heterogeneous catalysts for the preparation of primary and secondary amines from nitriles
INVENTOR(S): Pfeffinger, Joachim; Huellmann, Michael; Hoehn, Arthur; Funke, Frank; Ohlbach, Frank; Gerlach, Till
PATENT ASSIGNEE(S): BASF Aktiengesellschaft, Germany
SOURCE: Eur. Pat. Appl., 8 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1221437	A1	20020710	EP 2002-129	20020104
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
DE 10100313	A1	20020711	DE 2001-10100313	20010105
US 2002091194	A1	20020711	US 2002-33912	20020103
US 6790995	B2	20040914		
CN 1365965	A	20020828	CN 2002-104788	20020104
JP 2002226440	A2	20020814	JP 2002-609	20020107
PRIORITY APPLN. INFO.:			DE 2001-10100313	A 20010105
OTHER SOURCE(S):	MARPAT 137:80624			
REFERENCE COUNT:	4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT			

L3 ANSWER 7 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Secondary amines (XCH2)2NH [X = (un)substituted alkyl, cycloalkyl, dialkylamino, etc.; e.g., bis[3-(dimethylamino)]propylamine] are obtained from nitriles XCN [e.g., 3-(dimethylamino)propionitrile] by hydrogenation of the nitrile at 20-250°/60-350 bars in the presence of a hydrogenation catalyst containing 0.1-5% rhodium on a support (e.g., γ-alumina) so as to produce a mixture of the secondary amine as well as the corresponding primary amine XCHZNH2 [e.g., 3-(dimethylamino)propylamine] which is recycled back to the hydrogenation reaction.

ACCESSION NUMBER: 2002:517941 CAPLUS
DOCUMENT NUMBER: 137:80623
TITLE: Hydrogenation process and supported rhodium catalysts for the preparation of secondary amines from nitriles
INVENTOR(S): Pfeffinger, Joachim; Huellmann, Michael; Hoehn, Arthur; Funke, Frank; Ohlbach, Frank; Gerlach, Till
PATENT ASSIGNEE(S): BASF Aktiengesellschaft, Germany
SOURCE: Eur. Pat. Appl., 8 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1221436	A2	20020710	EP 2001-130515	20011221
EP 1221436	A3	20021120		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
DE 10100314	A1	20020711	DE 2001-10100314	20010105
US 2002128513	A1	20020912	US 2001-26900	20011227
US 6525223	B2	20030225		
CN 1367164	A	20020904	CN 2001-145301	20011231
JP 2002241351	A2	20020828	JP 2002-179	20020104
PRIORITY APPLN. INFO.:			DE 2001-10100314	A 20010105
OTHER SOURCE(S):	MARPAT 137:80623			

L3 ANSWER 8 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Regulatory guidelines for the assessment of acute dermal and ocular toxicity refer to the need to take the pH values of chems. into consideration, since the acidic and basic properties of chems. are known to play a role in the generation of acute dermal and ocular lesions. However, not all test guidelines provide an objective interpreting pH measurements in terms of acute skin or eye toxicity. The aim of this study was to develop classification models based on pH data for predicting the potential of chems. to cause skin corrosion, skin irritation and eye irritation. The possible application of these models in the context of tiered testing strategies is discussed.

ACCESSION NUMBER: 2001:856688 CAPLUS
DOCUMENT NUMBER: 136:212000
TITLE: The use of pH measurements to predict the potential of chemicals to cause acute dermal and ocular toxicity
AUTHOR(S): Worth, Andrew P.; Cronin, Mark T. D.
CORPORATE SOURCE: Joint Research Centre, Institute for Health and Consumer Protection, TP 580, ECVAM, European Commission, Ispra (VA), 21020, Italy
SOURCE: Toxicology (2001), 169(2), 119-131
CODEN: TXCYAC; ISSN: 0300-483X
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 9 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Some of the largest com. produced primary amines are manufactured by catalytic hydrogenation of nitriles using sponge metal catalysts. The larger the market volume for the amine, the more important the technol. used to control selectivity becomes to remain a viable producer. We have found that controlling the selectivity to the primary amine using lithium hydroxide modified sponge cobalt in backmix reactors, batch, semi-batch or continuous, at moderate pressures and temps. provides an excellent means of minimizing byproducts without sacrificing productivity. LiOH modified sponge cobalt was found to recycle in batch processing without loss of selectivity for primary amines. In continuous backmix processing LiOH modified sponge cobalt catalyst retained selectivity through numerous reactor turnovers compared to LiOH modified sponge nickel. NaOH and KOH modified catalysts tended to agglomerate under similar conditions. Procedures using a semi-batch system are provided for selecting optimum catalysts for nitrile hydrogenation, measuring the catalysts activity and its ability to resist poisoning by nitriles. This paper presents a practical approach to selecting the best selectivity control for the com. production of primary amines and demonstrates that chemical additives alone are not enough to allow one to obtain the best possible control over selectivity and in fact, the mode of operation and reaction conditions are also important in the optimization process.

ACCESSION NUMBER: 2001:439661 CAPLUS
DOCUMENT NUMBER: 136:120171
TITLE: Lithium hydroxide modified sponge catalysts for control of primary amine selectivity in nitrile hydrogenations
AUTHOR(S): Johnson, Thomas A.; Freyberger, Douglas P.
CORPORATE SOURCE: Consultant for Process Development Chemistry, Orefield, PA, 18069, USA
SOURCE: Chemical Industries (Dekker) (2001), 82(Catalysis of Organic Reactions), 201-227
CODEN: CHEIDI; ISSN: 0737-8025
PUBLISHER: Marcel Dekker, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 10 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Hydrogenation of 3-(dimethylamino)propionitrile over palladium catalysts was studied. Besides the expected amines, N,N,N',N'-tetramethylpropane-1,3-diamine, N,N-dimethyl-N'-propyl-propane-1,3-diamine, and N,N-bis[3-(dimethylamino)propyl]propylamine were found. Reaction pathways of their formation were discussed. Effects of reaction conditions, type of catalyst, and addition of ammonia or an amine into the charge on the hydrogenation selectivity were studied.

ACCESSION NUMBER: 2001:61467 CAPLUS
DOCUMENT NUMBER: 134:266016
TITLE: Hydrogenation of 3-(dimethylamino)propionitrile over palladium catalysts
AUTHOR(S): Krupka, Jiri; Pasek, Josef; Navratilova, Marketa
CORPORATE SOURCE: Department of Organic Technology, Institute of Chemical Technology, Prague, Prague, 166 28/6, Czech Rep.
SOURCE: Collection of Czechoslovak Chemical Communications (2000), 65(11), 1805-1819
CODEN: CCCCAK; ISSN: 0010-0765
PUBLISHER: Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:266016
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 11 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB The basic process comprises contacting the nitrile with H in the presence of a sponge or Raney Co catalyst and LiOH and H2O for conversion of the nitrile group to the primary amine. The conversion achieves high production rates of primary amine, high selectivity to primary amine thus avoiding secondary amine formation, eliminates the need for ammonia, and long catalyst life. The catalyst may be pretreated with the LiOH and/or the reaction may be carried out with the LiOH present in the reaction medium. Thus, 3-dimethylaminopropionitrile was hydrogenated utilizing a sponge cobalt catalyst and carrying out the reaction in the presence of a LiOH/water medium to 99.5% conversion in 1 and 1/2 h.

ACCESSION NUMBER: 1999:104538 CAPLUS
DOCUMENT NUMBER: 130:155254
TITLE: Hydrogenation catalyst modified with lithium hydroxide for conversion of nitriles to produce amines
INVENTOR(S): Johnson, Thomas Albert
PATENT ASSIGNEE(S): Air Products and Chemicals, Inc., USA
SOURCE: U.S., 12 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5869653	A	19990209	US 1997-960909	19971030
EP 913388	A1	19990506	EP 1998-120248	19981026
EP 913388	B1	20031001		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
BR 9804251	A	19991221	BR 1998-4251	19981027

PRIORITY APPLN. INFO.: US 1997-960909 A 19971030
REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 12 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB The title compound was prepared in 6 steps starting from acrylonitrile.

ACCESSION NUMBER: 1996:334271 CAPLUS
DOCUMENT NUMBER: 125:10213
TITLE: Preparation of 1-cyclohexyl-3-(γ-dimethylaminopropyl)carbodiimide methiodide
AUTHOR(S): Wen, Sujie; Zhu, Ning
CORPORATE SOURCE: Zhongnan Inst. New Technol. P.L.A., Yichang, 443200, Peop. Rep. China
SOURCE: Huaxue Shiji (1996), 18(1), 43-44
CODEN: HUSHDR; ISSN: 0258-3283
PUBLISHER: Huagongbu Huaxue Shiji Keji Qingbao Zhongxinzhan
DOCUMENT TYPE: Journal
LANGUAGE: Chinese

L3 ANSWER 13 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB RICN were transfer hydrogenated using R2CH2NH2 [R1, R2= alkyl, X(CH2)y, (CH2)kNMe2, (CH2)mPh, (CH2)nNH(CH2)n+1NH2, (CH2)pNH(CH2)pCN; x = cyano, H2NCH2; k = 2-17; m = 1-17; n, p = 3-11; yr = 3-16] at 20-200° in the presence of Raney Ni and in the absence of H. Thus, hexanenitrile (I) 3.2 g and octylamine (II) 3.1 g were heated at 100° with 3.4 g Raney Ni for 45 min to give a mixture containing I 31, II 48, hexylamine 8.1, and octylnitrile 2.7 area %.

ACCESSION NUMBER: 1994:30458 CAPLUS
DOCUMENT NUMBER: 120:30458
TITLE: Transfer hydrogenation of nitriles using amine donors
INVENTOR(S): Weigert, Frank J.
PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5237088	A	19930817	US 1992-857344	19920325
PRIORITY APPLN. INFO.:			US 1992-857344	19920325

OTHER SOURCE(S): CASREACT 120:30458; MARPAT 120:30458

L3 ANSWER 14 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Amines are prepared by hydrogenation of nitriles with metal catalysts prepared by mixing polyalc. solns. of salts of hydrogenating metals with metal alkoxides or silica sol (as materials for supports), treatment with H2O for hydrolysis, drying the resulting gels, optional calcining, and reduction Ni(NO3)2 was dissolved in ethylene glycol, treated with Et silicate at 80° for 3 h, and treated with H2O at 80° for 3 h to give gel, which was dried, calcined at 500° for 3 h, and reduced at 500° for 2 h under H to give Ni catalyst supported on silica. Autoclaving succinonitrile with ammonia and the catalyst at 100° and 20 atm H for 12 min gave 95% 4-aminobutyronitrile.

ACCESSION NUMBER: 1993:516785 CAPLUS
DOCUMENT NUMBER: 119:116785
TITLE: Preparation of amines by hydrogenation of nitriles
INVENTOR(S): Nakamura, Katsumi; Okamoto, Yasushi
PATENT ASSIGNEE(S): Nitto Chemical Industry Co Ltd, Japan
SOURCE: Jpn. Koksi Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05097776	A2	19930420	JP 1991-289429	19911009
JP 3014192	B2	20000228		
PRIORITY APPLN. INFO.:			JP 1991-289429	19911009

OTHER SOURCE(S): CASREACT 119:116785

L3 ANSWER 15 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Me2N(CH2)3NH2 (I) is prepared by hydrogenation of Me2NCH2CH2CN (II) in the presence of an alkaline earth oxide which suppresses formation of H2N(CH2)3NH2 (III). Thus, II, Raney Co, and NH3 (liquid) were charged to an autoclave and the whole maintained at 160° and 150 bar H to give I containing <50 ppm III.

ACCESSION NUMBER: 1989:614118 CAPLUS
DOCUMENT NUMBER: 111:214118
TITLE: Process for the preparation of N,N-dimethyldiaminopropane
INVENTOR(S): Kiel, Wolfgang; Bauer, Wolfgang
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Eur. Pat. Appl., 3 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 316761	A2	19890524	EP 1988-118720	19881110
EP 316761	A3	19900704		
R: CH, DE, FR, GB, LI				
DE 3739260	A1	19890601	DE 1987-3739260	19871118
PRIORITY APPLN. INFO.:			DE 1987-3739260 A	19871118

OTHER SOURCE(S): CASREACT 111:214118

L3 ANSWER 16 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Exptl. design data for the synthesis of N,N-dimethyl-1,3-propanediamine (I) by catalytic hydrogenation of β-dimethylaminopropionitrile (II) showed that optimum conversion of II at maximum I yield I can be obtained at H-II mol ratio ≥10 and 106-130°. In an exptl. verification of the process at H-II mol ratio 10 and 110°, II conversion was 95% and I yield was 91%.

ACCESSION NUMBER: 1989:576694 CAPLUS
DOCUMENT NUMBER: 111:176694
TITLE: Search for optimal conditions for synthesis of N,N-dimethyl-1,3-propanediamine by the experimental design method
AUTHOR(S): Popovich, O. T.; Pavlenko, N. V.; Golodets, G. I.
CORPORATE SOURCE: Kiev. Politekh. Inst., Kiev, USSR
SOURCE: Khimicheskaya Tekhnologiya (Kiev) (1989), (4), 58-61
CODEN: KHMTA6; ISSN: 0368-556X
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 111:176694

L3 ANSWER 17 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB The crosslinker diacrylyl-piperazine produces polyacrylamide gels with improved detection of proteins by ammoniacal Ag staining by reducing the background. The residual background staining caused by the catalytic reagents utilized in the polymerization of acrylamide gels was examined The commonly used catalyst system, tetramethyl-ethylenediamine and ammonium persulfate was responsible for the yellow staining background found after a prolonged development time with Ag staining. An alternate catalyst system has been designed to decrease further the formation of this background staining. Dimethyl-piperazine or tetramethylethylenediamine, K or ammonium persulfate, and Na thiosulfate are shown to provide for gels which have excellent mech. and staining characteristics. These catalytic systems produce little background staining despite prolonged development time with the ammoniacal Ag stain, and they reduce background staining with the dichromate Ag stain.

ACCESSION NUMBER: 1989:549870 CAPLUS
DOCUMENT NUMBER: 111:149870
TITLE: 'Catalysts' for polyacrylamide gel polymerization and detection of proteins by silver staining
AUTHOR(S): Hochstrasser, Denis F.; Merril, Carl R.
CORPORATE SOURCE: Clin. Neurogenet. Branch, Natl. Inst. Ment. Health, Bethesda, MD, 20892, USA
SOURCE: Applied and Theoretical Electrophoresis (1988), 1(1), 35-40
CODEN: ATELEM; ISSN: 0954-6642
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 18 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dissoln. rates were determined for S in 20-40 weight% aqueous NaOH(KOH) at 20-40° in the presence of added ethanolamines, polyamines, diethylene glycol, or glycerol. The organic additives exert a catalytic effect on S dissoln.

ACCESSION NUMBER: 1988:193464 CAPLUS
DOCUMENT NUMBER: 108:193464
TITLE: Dissolution of sulfur in aqueous solutions of alkali metal hydroxides in the presence of organic bases
AUTHOR(S): Fakhriev, A. M.; Mazgarov, A. M.
CORPORATE SOURCE: Vses. Nauchno-Issled. Inst. Uglevodorodn. Syr'ya, USSR
SOURCE: Zhurnal Prikladnoi Khimii (Sankt-Peterburg, Russian Federation) (1988), 61(1), 20-4
CODEN: ZPKHAB; ISSN: 0044-4618
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L3 ANSWER 19 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines were prepared by hydrogenation of nitriles using a Raney Co catalyst which contains from about 2 to 35 weight % Al on a 100 weight % total weight basis. The catalyst is prepared under low-temperature conditions from a Co-Al alloy. Thus, 3-(dimethylamino)propionitrile (I) was hydrogenated in the presence of a Raney Co-Al catalyst; at 65° and 500 psi H2 pressure, 100% reduction of I to the corresponding amine was obtained in 1.75 h.

ACCESSION NUMBER: 1983:178731 CAPLUS
DOCUMENT NUMBER: 98:178731
TITLE: Hydrogenation of nitriles to primary amines
INVENTOR(S): Allain, Ronald J.; Smith, Gerald D.
PATENT ASSIGNEE(S): Nalco Chemical Co. , USA
SOURCE: U.S., 9 pp. Cont. of U.S. Ser. No. 924,327, abandoned.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4375003	A	19830222	US 1979-3138	19790115
PRIORITY APPLN. INFO.:			US 1976-743731	A1 19761122
			US 1977-841501	A1 19771011
			US 1978-924327	A1 19780713

L3 ANSWER 20 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB The hydrogenation products of 3-dimethylaminopropionitrile were analyzed by gas chromatog. with a thermal conductivity detector. The analytes and capronitrile (internal standard) were separated at 70-170° on a 2-m + 3-mm column packed with Tsvetokhrom-1K treated with 2.5% KOH and 20% PEG-20M.

ACCESSION NUMBER: 1981:167151 CAPLUS
DOCUMENT NUMBER: 94:167151
TITLE: Chromatographic determination of products of 3-dimethylaminopropionitrile hydrogenation to N,N-dimethyl-1,3-propanediamine
AUTHOR(S): Rudaeva, E. G.; Ovchinnikova, V. P.; Makarovskii, I. A.; Yakushkin, M. I.
CORPORATE SOURCE: Vses. Nauchno-Issled. Inst. Neftekhim. Protsess., Leningrad, USSR
SOURCE: Khimicheskaya Promyshlennost, Seriya: Metody Analiza i Kontrolya Kachestva Produktsii v Khimicheskoi Promyshlennosti (1980), (12), 21-3
CODEN: KPSPDF
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L3 ANSWER 21 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Wastewaters from petrochem. plants and oil refineries contain 3-dimethylaminopropionitrile (I) [1738-25-6] and N,N-dimethyl-1,3-propanediamine (II) [109-55-7]. In relation to the purification of wastewaters containing these compds., concentrated solns. were obtained in the lab of I and II. The biol. oxidizability (BO) of these were determined as a ratio BOD20/COD. A biol. treatment scheme is described through the use of activated sludge in aeration lagoons. During the biodegrdn. of I and II, a significant increase was observed in the content of NH3-N which is used in the stabilization of the pH. The BO of I and II, over a wide pH interval, is related to the concentration of these compds. in the original wastewater. The maximum contents of I and II, not affecting the biol. oxidation under optimum technol. conditions, are, resp., 1300 mg/L (COD 4.46 g O2/L) and 800 mg/L (COD 2.14 g O2/L); after biol. oxidation, these 2 are reduced to 11.5 and 16.4 mg/L, resp.
ACCESSION NUMBER: 1981:162158 CAPLUS
DOCUMENT NUMBER: 94:162158
TITLE: Biological oxidizability of dimethylaminopropionitrile and dimethylpropanediamine in industrial wastewater
AUTHOR(S): Lisunova, T. S.; Karazeeva, L. N.; Krasnoborod'ko, L. A.
CORPORATE SOURCE: Vses. Nauchno-Issled. Inst. Neftekhim. Protsessov, Leningrad, USSR
SOURCE: Khimiya i Tekhnologiya Topliv i Masel (1980), (10), 54-6
CODEN: KTPMAG; ISSN: 0023-1169
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L3 ANSWER 22 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Dedeuteration of (MeCD2)2CO was studied in the presence of HClO4 NaOH and 4 amines. The Broensted β for the amines is 0.56. The monoprotonated forms of both Me2NCH2CR2CH2NH2 (R = H, Me) are bifunctional catalysts for the dedeuteration. Their primary-amino group transforms the ketone to an iminium ion, from which the tertiary-amino group removes D+ internally.
ACCESSION NUMBER: 1980:638196 CAPLUS
DOCUMENT NUMBER: 93:238196
TITLE: Catalysis of α-hydrogen exchange. 21. Stereoselective bifunctional catalysis of the dedeuteration of 3-pentanone-2,2,4,4-d4
AUTHOR(S): Hine, Jack; Zeigler, James P.
CORPORATE SOURCE: Dep. Chem., Ohio State Univ., Columbus, OH, 43210, USA
SOURCE: Journal of the American Chemical Society (1980), 102(25), 7524-9
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 23 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB The heats of combustion, formation, and evaporation (kcal/mol) of the title compds. were, resp.: EtCONMe2, 773.57, -59.79, 12.64; PrCONMe2, 930.44, -64.74, 13.19; Me(CH2)7CONMe2, 1712.50, -89.38, 18.30; H2N(CH2)3NMe2, 820.8, 21.6, 12.68; Me2NCH2CH2CN, 926.5, -9.2, 11.30.
ACCESSION NUMBER: 1980:58010 CAPLUS
DOCUMENT NUMBER: 92:58010
TITLE: Heats of combustion of dimethyl-substituted amides and dimethylalkylamines
AUTHOR(S): Vasil'eva, T. F.; Kotov, V. I.
CORPORATE SOURCE: Vses. Nauchno-Issled. Inst. Neftekhim. Protsessov, Leningrad, USSR
SOURCE: Vses. Konf. Kalorim., [Rasshir. Tezisy Dokl.], 7th (1977), Volume 1, 102-6. Akad. Nauk SSSR, Inst. Khim. Fiz.: Moscow, USSR.
CODEN: 41VFAB
DOCUMENT TYPE: Conference
LANGUAGE: Russian

L3 ANSWER 24 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Title only translated.
ACCESSION NUMBER: 1979:22066 CAPLUS
DOCUMENT NUMBER: 90:22066
TITLE: Enthalpies of burning and formation of some dimethyl-substituted nitrogen-containing compounds
AUTHOR(S): Vasil'eva, T. F.; Zhil'tsova, E. N.; Dogonina, M. D.; Vasil'ev, I. A.; Kotov, V. I.
CORPORATE SOURCE: USSR
SOURCE: Termodinam. Organ. Soedin. (1977), (6), 41-5
From: Ref. Zh., Khim. 1978, Abstr. No. 13B799
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L3 ANSWER 25 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Title only translated.
ACCESSION NUMBER: 1979:22062 CAPLUS
DOCUMENT NUMBER: 90:22062
TITLE: Vapor pressure and enthalpies of vaporization of some dimethyl-substituted nitrogen-containing compounds
AUTHOR(S): Vasil'eva, T. F.; Petrov, V. M.
CORPORATE SOURCE: USSR
SOURCE: Termodinam. Organ. Soedin. (1977), (6), 74-6
From: Ref. Zh., Khim. 1978, Abstr. No. 13B787
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L3 ANSWER 26 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB The U.S. Occupational Safety and Health Administration issued a Health Hazard Alert for NIAX Catalyst ESN [62765-93-9] and its components dimethylaminopropionitrile (I) [1738-25-6] and bis(2-(dimethylamino)ethyl)ether [3033-62-3]. The catalyst is apparently neurotoxic and produces urinary bladder dysfunction as the prevailing clin. feature. Exposure may occur by inhalation, skin absorption, and ingestion. The catalyst is used in the manufacture of flexible polyurethane foams. I is also used as a component of acrylamide-based gels (for soil grouting, etc.) and as an intermediate in the synthesis of dimethylaminopropylamine [109-55-7] and some pharmaceuticals.
ACCESSION NUMBER: 1978:620132 CAPLUS
DOCUMENT NUMBER: 89:220132
TITLE: Current Intelligence Bulletin: NIAX catalyst ESN. A mixture of dimethylaminopropionitrile and bis[2-(dimethylamino)ethyl] ether
CORPORATE SOURCE: National Institute for Occupational Safety and Health, Cincinnati, OH, USA; Occupational Safety and Health Administration
SOURCE: DHEW (NIOSH) Publication (United States) (1978), 78-157, 1-3
CODEN: DNPUDQ
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 27 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Bifunctional catalysis of the dedeuteration of acetone-d6 by the monoprotonated forms of 9 primary-tertiary diamines is examined. Each catalyst uses its NH2 group to transform the ketone to an iminium ion from which D is removed internally by the Me2N group to give an enamine with a-NDMe2+ substituent. The D is then removed from the -NDMe2+ group by attack of a base. Rate consts. are determined for the formation of the iminium ions; rate and equilibrium consts. for the formation of enamines and rough relative rate consts. for removal of D from the -NDMe2+ groups are also determined. In the case of the eight 1,3-diamines studied, the transition state for enamine formation is most stable relative to acetone-d6 and monoprotonated diamine when there is eclipsing around the bond between the C atom to which the NH2 group is attached and the adjacent C atom, especially when the C-NH2 bond is eclipsed with the C-CNMe2 bond. Bifunctional catalysis is also observed with monoprotonated o-(dimethylaminomethyl)benzylamine; it is less effective than it would otherwise be because the reactant is stabilized by simultaneous coordination of the added H+ with both amino groups. Such internal H bonding in a monoprotonated diamine, which makes N,N,N',N',2,2-hexamethyl-1,3-propanediamine and o-bis(dimethylaminomethyl)benzene more basic than they would otherwise be, does not increase the rate at which such diamines remove D from acetone-d6 and hence results in large deviations from the Broensted equation for the monofunctional base-catalyzed reaction.
ACCESSION NUMBER: 1976:493386 CAPLUS
DOCUMENT NUMBER: 85:93386
TITLE: Catalysis of α-hydrogen exchange. XIX. Bifunctional catalysis of the dedeuteration of acetone-d6 by conformationally constrained derivatives of N,N-dimethyl-1,3-propanediamine
AUTHOR(S): Hine, Jack; Li, Wu-Shyong
CORPORATE SOURCE: Dep. Chem., Ohio State Univ., Columbus, OH, USA
SOURCE: Journal of the American Chemical Society (1976), 98(11), 3287-94
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 28 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Procedures have been developed for the preparation of nitrogen mustard derivs. containing amino, dimethylamino, and trimethylammonio groups separated from the mustard group by 2-, 3-, 4-, and 5-carbon chains. A β-trimethylammonio group diminished the reactivity of an amino group so that it was possible to introduce only 1 hydroxyethyl group by reaction with ethylene oxide. Biol. tests indicated the amino mustards to have toxic and antitumor properties similar to HN-2. The ammonio mustards were devoid of antitumor activity and were much less toxic.
ACCESSION NUMBER: 1965:462834 CAPLUS
DOCUMENT NUMBER: 63:62834
ORIGINAL REFERENCE NO.: 63:11467g-h
TITLE: Some amino and ammonio nitrogen mustard analogs
AUTHOR(S): Price, Charles C.; Kabas, Guglielmo; Nakata, Isao
CORPORATE SOURCE: Univ. of Pennsylvania, Philadelphia
SOURCE: Journal of Medicinal Chemistry (1965), 8(5), 650-5
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 29 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Keeping 100 ml. 22% aqueous Me2NH. 46 ml. 35% formalin, 0.1 ml. 5N NaOH and 41
ml. Me2C(OH)CN 3 hrs. gave after extraction with CHCl3, 55.2% Me2NCH2CN, b. 133-6°. Similarly was prepared 82.7% (CH2)5NCH2CN, b12 83-4° [(CH2)5N = piperidino]. CH2:CHCN and 22% aqueous Me2NH overnight gave 74.8% Me2NCH2CH2CN, b. 171-2°. Reduction of the nitriles with LiAlH4 in Et2O 2 hrs. gave: 65% Me2NCH2CH2NH2, b. 103-5°; and 62% (CH2)5 NCH2CH2NH2., b30 78-80°. Me2N(CH2)3NH2, 63%, b. 136-7°; Et2N(CH2)3NH2, 67.2%, b25 72°; and (CH2)5NCH2CH2CH2NH2, 81%, b8 80° were prepared by hydrogenation over Raney Ni at 80-100° under 100-20 atmospheric in MeOH-NH3. NaHSO3.CH2O treated with the above amines in H2O, the mixts. kept 1 hr., then treated with aqueous KCN 2 hrs., gave the following R2N(CH2)nNHCH2CN (R2N and n shown): Me2N, 2, 35.3%, b40 119°; Et2N, 2, 44%, b38 137-40°; (CH2)5N, 2, 32%, b6 118-19°; Me2N, 3, 40.6%, b5 104-5°; Et2N, 3, 51%, b4 114°; (CH2)5N, 3, 49%, b2 122-4°. These treated with dry N oxides in Et2O with cooling 2 hrs. (until blue-green color had formed) gave an oily precipitate which with Et2O.HCl gave the following 3-dialkyl-aminoalkylsydnone imines (I) (R and n shown), isolated as di-HCl salts: Me, 2, m. 165-6°; Et, 2, m. 151°; (R2N =)(CH2)5N, 2, m. 162-3°; Me, 3, m. 170-1°; Et, 3, m. 162-3° (isolated as picrate); (R2N=)(CH2)5N, 3, m. 156-7°.
ACCESSION NUMBER: 1963:403479 CAPLUS
DOCUMENT NUMBER: 59:3479
ORIGINAL REFERENCE NO.: 59:602f-h,603a
TITLE: Sydnones and sydnone imines. XV. Synthesis of 3-(dialkylaminoalkyl)sydnone imines
AUTHOR(S): Yashunskii, V. G.
CORPORATE SOURCE: S. Ordzhonikidze All-Union Chem.-Pharm. Res. Inst., Moscow
SOURCE: Zhurnal Obshchei Khimii (1963), 33, 192-5
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 30 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. CA 55, 22328i; Elslager, et al., CA 51, 1182d. Several dialkylaminoalkylaminopyridines and pyrimidines were prepared N-Cyanoethylation of the corresponding amines gave N-eyanoethylpiperidine, b. 210°, in 94% yield and Et2NCH2CN, b. 170°, in 80% yield. Gradual addition of 25% Me2NH solution to CH2:CHCN gave 86% Me2NCH2CH2CN, b. 171-2°; picrate m. 155°. Et2-NCH2CH2CN, b. 195-6°, 92%, piperidinepropionitrile, b. 220-2°, 90%, and morpholinopropionitrile, b. 244-6°, 90% yield, were prepared by the method of Whitemore, et al., (CA 38, 36173). Nitriles were reduced with Raney Ni in slightly alkaline solns. (e.g. 0.1 g. NaOEt/0.1 mole nitrile) to amines: β-piperidinoethylamine, b. 184°, 70% yield; Et2NCH2CH2NH2, b. 145°, 65% yield; Me2NCH2CH2CH2-NH2, b. 125-6°, 65% yield (picrate m. 220°); Et2NCH2CH2-CH2NH2, b. 168°, 78% yield; β-piperidinopropylamine, b. 202-4°, 85% yield; γ-morpholinopropylamine, b. 216-18°, 88% yield. AcCH2CO2Et (13 g.) and NH2CSNH2 (18 g.) were added to 3 g. Na in 50 ml. alc., the mixture kept 1 hr. at 50°, refluxed 2 hrs., the alc. distilled, the residue dissolved in water, and acidified with AcOH to give 4-methyl-2-thiouracil (95% yield), m. above 270° (AcOH). This in 5% NaZCO3 was treated with Me2SO4 to give 4-methyl-2-methylthiouracil, m. 217-19°. 2-Chloro-5-nitropyridine refluxed in alc. with fused NaOAc and the appropriate amine gave the following 5-nitro-2-(γ-dialkylaminopropylamino)-pyridines (dialkylamino group, % yield, m.p., m.p. of picrate given): Me2N, 70, 64°, --; Et2N, 72, 78-80°, --; piperidino, 70, 80-2°, 195°; morpholino, 75, 102°, 112°. (These compds. were hygroscopic, m.ps. were determined in sealed tubes.) Heating substituted 2-methylthiopyrimidines with the appropriate amine at 170 gave the following 6,4-R'-(HO)C4N2NH(CH2)nR''-2 (n, R', R'', % yield, m.p., m.p. of picrate given): 1, Me, Ph, 55, above 250°, 190°; 1, OH, Ph, 45, above 250°, 204°; 2, Me, OH, 95, 190°, 194°; 2, OH, OH, 80, 172°, 174°; 2, Me, piperidino, 75, above 240°, 175°; 2, Me, Et2N, 80, above 250°, 244°; 3, Me, Me2N, 65, 68°, 178°; 3, Me, Et2N, 70, 70°, 193°; 3, Me, piperidino, 80, 75°, 210°; 3, Me, morpholino, 75, 98°, 218°. Treating the appropriate 2-dialkylaminoalkylaminopyrimidine in C6H6 with Cl2CHCOCl gave the following 6,4-Me-(HO)C4N2[N(COCHCl2)(CH2)nR]-2 (n, R, % yield, m.p. given) (recrystd. from dimethylformamide): 1, Ph, 40, 190°; 2, OH, 55, 172°; 2, piperidino, 56, 134°; 3, Et2N, 60, 84°; 3, morpholino, 58, 98°; 3, piperidino, 62. 86°. The appropriate alc. treated with SOCl2 in C6H6 gave the following ethyl chloride hydrochlorides: 2-piperidino, m. 226°; 2-morpholino, m. 180°. Refluxing 2-(2-hydroxy-ethylamino)-4-methyluracil in C6H6 with NaNH2 and the appropriate dialkylaminoethyl chloride HCl gave the following 6,4-R'(HO)C4N2[N(CH2CH2Cl)CH2CH2R'']-2 (R', R', % yield, m.p. given): OH, OH, 50, 181°; Me, OH, 50, above 270°; Me, Et2N, 55, 198° (hygroscopic); Me, piperidino, 60, 204° (hygroscopic); Me, morpholino, 60, 217° (hygroscopic).
ACCESSION NUMBER: 1962:423227 CAPLUS
DOCUMENT NUMBER: 57:23227
ORIGINAL REFERENCE NO.: 57:4662a-g
TITLE: Possible antiamebic agents. XVI
AUTHOR(S): Sen, A. B.; Gupta, S. K.
CORPORATE SOURCE: Univ. Lucknow, India
SOURCE: J. Indian Chem. Soc. (1962), 39, 129-34
DOCUMENT TYPE: Journal

L3 ANSWER 30 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
LANGUAGE: Unavailable

L3 ANSWER 31 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB R2MeNI(CH2)nNHC(:NH)NH2.HI (I) were prepared, where R was an alkyl radical or R2N a heterocyclic radical and n = 2-5. To 350 cc. com. aqueous NaHSO3 and 100 cc. 40% aqueous CH2O was added gradually 1 mole amine, 1st at 65° and then at 35° (cooling) with stirring and under reflux (with highly volatile amines) (the com. aqueous solns. or the anhydrous amines could be used), the mixture treated during 90 min. with 150 cc. 50% aqueous NaCN, and the upper nitrile layer decanted, dried, and distilled to give the following R2N(CH2)nCN (II) (n = 1) (R, % yield, b.p./ mm., m.p. of methiodide given): Me, 71, 138°/.apprx.760, 210°; Et, 67 64°/15, 181°; (R2N =) pyrrolidino, 60, 84-5°/17, 216°; (R2N =) piperidino, 72.5, 95°/15, 197°. CH2:CHCN (III) (equimolar amount) added gradually to a secondary amine (com. aqueous solution or anhydrous diluted with C6H6) below 30°, the mixture stirred 2 hrs., and the nitrile separated by distillation (the nitriles were salted out when present in aqueous solution, dried, and distilled) gave the following II (n = 2) (R, solvent, % yield, and b.p./mm. given): Me, H2O, 90, 72°/19 (HCl salt m. 203°); Et, H2O, quant., 89-90°/16 (HCl salt m. 126°); (R2N =) pyrrolidino, C6H6, quant., 104-5°/20 (methiodide m. 126°); (R2N =) piperidino, C6H6, 96%, 110-11°/16 (methiodide m. 156-7°). γ-Butyrolactone (1 mole), 50 cc. MeOH, and an unsealed ampul containing 80 cc. liquid NH3 placed in a 500 cc. steel autoclave, the contents stirred vigorously, heated 16 hrs. at 100° (bath temperature), cooled, filtered, the filtrate evaporated in vacuo, the residue treated with 80 cc. C6H6, and the mixture evaporated on a H2O bath gave 97 g. crude HO(CH2)3CONH2 (IV). Crude IV (51 g.) in 100 cc. CHCl3 treated gradually with 130 g. SOCl2 (highly exothermic reaction), when the reaction subsided the solution boiled until evolution of HCl ceased, and distilled gave 36 g. Cl(CH2)3CN (V), b15 81°. The anhydrous secondary amines (2 moles) and 1 mole V in Me2CO heated 24-48 hrs. at 100° in an autoclave, the precipitate filtered off, and the filtrate fractionated (in the case of pyrrolidine where its HCl salt was soluble in Me2CO, the Me2CO was removed on a H2O bath, the base was liberated with alkali, decanted, and distilled) gave the following II (n = 3) (R, % yield, b.p./mm., m.p. of methiodide given): Me, 78, 91-2°/18, 203°; Et, 70, 97°/18, 193°; (R2N =) pyrrolidino, 78, 115°/18, 143°; (R2N =) piperidino, 80, 126°/18, 124°. Pyrolysis of MeCH:CHCH(CN)OBz at 450 ± 10° (method of Snyder et al., CA 43, 4217g) gave 77% Me2NCH2CH:CHCH2CN, b32 53°. Me2NCH2CH2OH (0.33 mole), 50 cc. C6H6, and 30 drops 40% aqueous Triton B treated gradually with III with stirring below 25°, the mixture stirred 2 hrs., neutralized with 2 g. NH4Cl, filtered, and the filtrate distilled gave 90% R2NCH2CH2OCH2CH2CN (VI) (R = Me), b18 114-15°; methiodide m. 125°. Similarly was prepared 92% VI (R = Et). The preceding nitriles were reduced (A) chemical with Na in EtOH-PhMe (method of Bloom, et al., CA 39, 24869) and (B) catalytically (1) in MeOH solution at 90-100° with Raney Co and liquid NH3, (2) in MeOH solution at 90-100° with Raney Ni, and (3) in MeOH solution at 60° with Raney Ni to give the following R2N(CH2)nNH2 (VII) [R, n, method, initial pressure (kg./cm.), % yield, b.p. given]: Me, 2, A, -, -, 108°; Et, 2, A, -, 46, 145°; (R2N =) pyrrolidino, 2, A, -, 43,

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184-5°; (R2N =) piperidino, 2, A, -, 45, 134-5°; Me, 3, B-1, 130, 53, 168°; Et, 3, B-1, 110, 85, 167°; (R2N =) pyrrolidino, 3, B-1, 75, 64, 187°; (R2N =) piperidino, 3, B-1, 90, 69, b18 89-90°; Me, 4, B-1, 70, 50, 157°; Et, 3, B-1, 72, 59, 189°; (R2N =) pyrrolidino, 4, B-1, 70. 64, 205° (b19 95-7°); (R2N =) piperidino, 4, B-1, 80, 73, 224-5°; Me, 5, B-2, 80, 39, b16 79-80°; Et, 5, B-2, 80, 43, b16 10.3°; and the following R2NCH2CH2O(CH2)3NH2: Me, -, B-3, 70, 52, b23 99-100°; Et, -, B-3, 75, 59, b20 112-13°. [MeSC(:NH)NH2]2.H2SO4 (0.5 mole), 1.1 moles NaI, and 250 cc. abs. EtOH refluxed 4 hrs., filtered, the filtrate evapd., the residue treated with 100 cc. Me2CO, the mixt. filtered, the soln. evapd., and the product washed with cold EtOAc gave 88% MeSC(:NH)NH2.HI (VIII), m. 117°. VII (R = Me, n = 2) (IX) HCl salt (12 g.) and 16.2 g. VIII added to NaOEt soln. (from 3.5 g. Na and 75 cc. EtOH), the mixt. refluxed 45 min., evapd., the residue dissolved in 40 cc. Me2CO, the filtered soln. dild. with an equal vol. of BuOH, and treated gradually with 10.5 g. MeI with cooling gave I (R = Me, n = 2) (X), m. 181° (Me2CO-MeOH). VII (R = Et, n = 2) (0.1 mole) and 0.1 mole VIII in 60 cc. abs. EtOH refluxed until MeSH ceased to evolve, the EtOH evapd. in vacuo on a H2O bath, the residue taken up in 50 cc. Me2CO, the filtered soln. cooled, and treated gradually with 0.1 mole MeI gave I (R = Et, n = 2), m. 159° (EtOAc-MeOH). The following I were prepd. by the latter method in 60-80% yields (R, n, and m.p. given): (R2N =) pyrrolidino, 2, 136°; (R2N =) piperidino, 2, 136°; Me, 3, 153-4°; Et, 3, 151°; (R2N =) pyrrolidino, 3, 121°; (R2N =) piperidino, 3, 158°; Me, 4, 171.5°; Et, 4, 115.5°; (R2N =) pyrrolidino, 4, 131°; (R2N =) piperidino, 4, 156-7°; Me, 5, -; Et, 5, 138-9°. R2MeNICH2CH2O-(CH2)3NHC(:NH)NH2.HI (XI) (R = Me), m. 110°, and XI (R = Et) (dipicrate), were also prepd. Proof of structure of the I. Application of the Sakaguchi reaction to the I gave a pos. reaction, which did not occur with a mono-substituted guanidine. To a concd. soln. of 0.1 mole BrCH2CH2NH2.HBr in MeOH was added 0.3 mole anhyd. Me3N (previously chilled), the ppt. collected, the filtrate evapd. in vacuo, the residual basic oil dissolved in MeOH-iso-PrOH, the soln. neutralized with HBr, and the product dried to give Me3NBrCH2CH2NH2.HBr (XII). XII dissolved in a suspension of moist Ag2O (from 0.2 mole AgNO3) in H2O, the mixt. stirred several min., filtered, the filtrate neutralized with HI, evapd. in vacuo, and the residue washed with iso-PrOH-Me2CO gave Me3NICH2CH2NH2.HI (XIII). XIII (0.05 mole) added to NaOEt soln. (from 0.05 mole Na and 75 cc. abs. EtOH), the soln. treated with 0.05 mole VIII, refluxed 2 hrs., and evapd. to 1/3 vol. gave X, m. 181° (iso-PrOH-MeOH), identical with X prepd. above. To 0.5 mole MeNHCSNH2 in 150 cc. Me2CO was added gradually 0.5 mole MeI with stirring to give 93% MeSC(:NH)NH-Me.HI (XIV), m. 135° (Me2CO). XIV (0.05 mole) and 0.05 mole IX in 30 cc. EtOH refluxed 30 min., cooled, treated with 0.05 mole HI (as 66% soln.), and dild. with EtOAc gave 94% Me2N(CH2)nNHC(:NH)NHMe.2HI (XV) (n = 2), m. 142-3° (EtOH-iso-PrOH). Similarly was prepd. XV (n = 3), m. 121-2°.

ACCESSION NUMBER: 1962:31056 CAPLUS
DOCUMENT NUMBER: 56:31056
ORIGINAL REFERENCE NO.: 56:5830d-i,5831a-h
TITLE: Preparation of guanidines having in addition a quaternary ammonium function
AUTHOR(S): Lespagnol, A.; Cheymol, J.; Cuingnet, E.; Debaert, M.; Adolphe, M.; Adolphe, C.
CORPORATE SOURCE: Univ. Lille, Fr.
SOURCE: Congr. Sci. Pharm. (1960), 1959, 194-308
DOCUMENT TYPE: Journal

L3 ANSWER 31 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
LANGUAGE: French

L3 ANSWER 32 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Heating [in ethylene glycol (I)] of N-mono- and N,N-biscyanoethylated amino acids, which did not cyclize in the glycol, and also of some N-cyanoethylated amines, resulted in decyanoethylated products corresponding to the amino acids and amines. To 2.2 g. N-cyanoethyltaurine was added 13.2 g. (6-fold amount) of I, the mixture heated 4-5 hrs. at 160-70°, and the solution concentrated in vacuo and cooled to yield 1.3 g. taurine, decomposing 305-10°. Decyanoethylation of N, N-biscyanoethyltaurine, N-cyanoethyl-α-aminophenylacetic acid, N-cyanoethylphenylalanine, N-mono- and N,N-biscyanoethyltyrosine, and N-cyanoethyltriphenylmethylamine was carried out in the same manner. β-Diethylaminopropionitrile (14 g.) was heated with a 6-fold amount of I (4-5 hrs. at 190-200°) and the mixture distilled to yield small amts. of Et2NH; HCl salt m. 228-30°. β-Dimethylamino-propionitrile, β-(1-pyrrolidyl)propionitrile, β-piperidinopropionitrile, and β-morpholinopropionitrile were decyanoethylated in the same manner.

ACCESSION NUMBER: 1961:65072 CAPLUS
DOCUMENT NUMBER: 55:65072
ORIGINAL REFERENCE NO.: 55:12411a-c
TITLE: Decyanoethylation of N-cyanoethylated compounds
AUTHOR(S): Butskus, P. F.
CORPORATE SOURCE: State Univ., Vilnyus
SOURCE: Izvestiya Vysshikh Uchebnykh Zavedenii, Khimiya i Khimicheskaya Tekhnologiya (1960), 3, 1108-9
CODEN: IVUKAR; ISSN: 0579-2991
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 33 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Tertiary aminoacetonitriles were prepared by heating 350 ml. 30% NaHSO3 and 100 ml. 40% HCHO to 60°, cooling to 35°, dropping in 1 mole appropriate amine (40% in H2O), stirring 2 hrs., adding 150 ml. 50% NaCN, stirring 1.5 hrs., and drying the nitrile carefully with K2CO3 before distillation [b.p./mm., % yield, and m.p. (decomposition) of the methiodide given]: Me2NCH2CN, 138°/760, 71, 210°; Et2NCH2CN, 64°/15, 67, 181°; pyrrolidinoacetonitrile, 84-5°/17, 60, 216°; piperidinoacetonitrile, 95°/15, 72.5, 197°. The following propionitriles were prepared by stirring the amine in a solvent with CH2:CHCN below 30° [substituent, b.p./mm., % yield, solvent, and m.p. (decomposition) of salt given]: 3-dimethylamino, 72°/19, 90, H2O, 203° (HCl salt); 3-diethylamino, 89-90°/16, 100, H2O, 126° (HCl salt); 3-pyrrolidino, 104-5°/20, 100, C6H6, 126° (MeI salt); 3-piperidino, 96, C6H6, 156-7° (MeI). The nitriles were reduced with Na and EtOH (A) or catalytically with anhydrous Raney Co (A1) or Raney Co in liquid NH3 (B) to give the following amines [b.p., method, % yield given]: 2-dimethylaminoethylamine (I), 108°, A1, 65; 2-diethylaminoethylamine (II), 145°, A1, 72; 2-pyrrolidinoethylamine, 168°, A, 43; 2-piperidinoethylamine, 184-5°, A, 45; 3-dimethylaminopropylamine (III), 134-5°, B, 53; 3-diethylaminopropylamine (IV), 167°, B, 85; 3-pyrrolidinopropylamine, 187°, B, 64; 3-piperidinopropylamine, 89-90°/18 mm., B, 69. I (4.4 g.) boiled with 30 ml. absolute EtOH and 11 g. NH:C(SMe)NH2.HI, m. 117° (88% yield from the sulfate by refluxing 4 hrs. with NaI in absolute EtOH), until evolution of MeSH ceased, evaporated, the mixture dissolved in 40 ml. Me2CO and 40 ml. BuOH and 7 g. MeI added dropwise yielded 80% IMe3N(CH2)2R.HI (R = guanidino) (V), m. 181°. Similarly, III gave IMe3N(CH2)3R (VI), m. 153-4°. The following were prepared similarly but the reaction mixture was evaporated in vacuo and the MeI added to a Me2CO solution without BuOH: IMeEt2N(CH2)2R.HI (VII), m. 159°; 2-(N-methylpyrrolidino)-1-guanidinoethane-HI iodide, m. 136°; 2-(N-methylpiperidino)-1-guanidinoethane-HI iodide, m. 158°; IMeEt2N(CH2)3R.HI (VIII), m. 151°; 3-(N-methylpyrrolidino)-1-guanidinopropane-HI iodide, m. 121°, yield 78%; 3-(N-methylpiperidino)-1-guanidinopropane-HI iodide, m. 158°, yield 82%. III (11 g.), 50 ml. absolute EtOH, and 26 g. NH:C(SMe)NHMe.HI m. 135° (93% yield from MeNHCSNH2 and MeI in anhydrous Me2CO) refluxed 45 min., evaporated, Me2CO and MeI added yielded 78% IMe3N(CH2)3NHC(:NH)NHMe.HI m. 132°. Similarly, II and IV yielded 54% IMeEt2N(CH2)2NHC(:NH)NHMe.HI m. 154°, and IMeEt2N(CH2)3NHC(:NH)NHMe.HI m. 137°. Br(CH2)2NH2.HBr (20.9 g.) and 17.7 g. Me3N in a little MeOH gave [Me3N(CH2)2NH2]Br.HBr which was converted to the iodide with AgOH and HI. The iodide with NH:C(SMe)NH2.HI and NaOEt gave V, m. 181°. I (4.9 g.) refluxed 30 min. with NH:C(SMe)NHMe.HI and 30 ml. EtOH and 5.5 ml. HI (66%) added yielded 94% Me2N(CH2)2NHC(:NH)NHMe.2HI (IX), m. 142-3°. Similarly, III gave Me2N(CH2)3NHC(:NH)NHMe.2HI (X), m. 121-2°. VI, VII, and VIII gave colored Sakaguchi reactions indicating a monosubstituted guanidine, while the disubstituted guanidines IX and X did not give this reaction. The compds. contained groups with hypoglycemic properties.

ACCESSION NUMBER: 1961:17928 CAPLUS
DOCUMENT NUMBER: 55:17928
ORIGINAL REFERENCE NO.: 55:3584h-i,3585a-f
TITLE: Monosubstituted guanidines with a quaternary ammonium group

L3 ANSWER 33 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
AUTHOR(S): Lespagnol, Albert; Cuingnet, Etienne; Debaert, Michel
CORPORATE SOURCE: Fac. med. pharm., Lille, Fr.
SOURCE: Bulletin de la Societe Chimique de France (1960), 2, 383-9
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 55:17928

L3 ANSWER 34 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Compds. of the type RCH2NH(CH2)3R' (I), where R is substituted phenyl and R' is disubstituted amino, were prepared by condensation of 1) substituted benzyl bromides with γ-dialkylaminopropylamines or 2) equimolar amts. of substituted benzaldehydes with the same amines, followed by catalytic hydrogenation of the Schiff bases (EtOH-PtO2). Piperidine (40 g.) added to 50 g. cold CH2:CHCN, the mixture refluxed 1 hr. on a H2O bath, allowed to stand overnight at room temperature, then distilled gave 97% β-piperidinopropionitrile (II), b30 128-30°. Similarly prepared were 93% β-morpholino- and 94% β-diethylaminopropionitriles, b2 120-1°, and b760 196-8°, resp. II (51 g.) was reduced 3-4 hrs. at 500-550 lb./sq. in. H in a solution of 250 g. absolute MeOH previously saturated in the cold with about 90 g. dry NH3, the mixture filtered, about 25-30 ml. of filtrate distilled to remove NH3, the residue chilled, saturated with dry HCl, the MeOH distilled, the residual paste decomposed by slow addition of 70% NaOH until 2 liquid layers separated The upper was dried over KOH, then over Na wire and distilled, giving 72.4% γ-piperidinopropylamine (III), b1 88°; picrate, m. 208-9° (decomposition). Similarly prepared were 65% γ-morpholino-(IV), b1 92-3° (picrate, m. 165°), and 61% γ-diethylaminopropylamines, b. 170-5° (picrate, m. 195-6°). Addition of 3.6 g. 2,4-Cl2C6H3CH2Br to 6.3 g. III, the mixture stirred 1 hr., allowed to stand overnight, poured into a large volume of H2O, the semisolid extracted with CHCl3, the extract dried over anhydrous K2CO3, the solvent removed and the residue distilled gave 3.5 g. I (R = 2,4-Cl2C6H3, R' = C5H10N) (V), b2 193-5°. o-ClC6H4CHO (2.82 g.) was heated in vacuo on a steam bath 3 hrs. with 3.25 g. IV, the Schiff base dissolved in 50 ml. hot EtOH, the hot solution hydrogenated 3 hrs. at 60 lb./sq. in. over 0.25 g. PtO2, the mixture filtered, the filtrate concentrated and the residue distilled to give 5 g. I (R = 2-ClC6H4, R' = C4H8ON), b2 192-3°. Similarly prepared were the following I (R', % yield, b.p./mm. and m.p. of picrate given). For R = 2-ClC6H4: C5H10N, 62, 183-4°/2, 200-1°; C4H8ON, 91, 192-3°/2, 203-4°; Et2N, 77, 166-7°/2, 143-4°. For R = 4-ClC6H4: C5H10N, 58, 193-5°/2, 133-4°; C4H8ON, 71, 207-9°/2, 128-9°; Et2N, 65, 169-70°/2, 130-1°. For R = 2,4-Cl2C6H3: C5H10N, 64, 195-7°/7, 180-1°; C4H8ON, 76, 163-5°/2, 188-9°; Et2N, 93, 181-2°/3, 133-4°. For R = 2,5-HO(Cl)C6H3: C5H10N, 55,-(m. 137°), 188-9°; C4H8ON, 61, -(m. 140°), 208-9°, Et2N, 58, -(m. 109°), 168-9°. For R = 3,4-(MeO)2C6H3: C5H10N, 72, 218-20°/20, 195-6°; C4H8ON, 68, 250-2°/20, 222°; Et2N, 92, 173-4°/1, 152-3°. Cl2CHCOCl (0.75 g.) in 3 ml. dichloroethane (VI) added slowly to 1.5 g. V in 10 ml. VI containing 5 ml. N NaOH (all below 0°), the mixture allowed to warm to room temperature, the organic layer separated, washed with N NaOH, H2O, N HCl, H2O, the solution dried over K2CO3, the solvent removed and the residue crystallized (absolute EtOH petr. ether) gave 1.1 g. (53.5%) RCH2N(COCHCl2)(CH2)2R' (VII) (R = 2,4-Cl2C6H3, R' = C5H10NCH2), m. 163-4°. Similarly prepared were the following VII (R, R', % yield and m.p. given): 4-ClC6H4, C5H10NCH2, 53, 111°; 2,4-Cl2C6H3, C4H8ONCH2, 36.2, 173°; 2,4-Cl2C6H3, Et2NCH2, 40, 139°; 5,2-Cl(HO)C6H3, HO, 43, 147°.
ACCESSION NUMBER: 1959:83420 CAPLUS

L3 ANSWER 34 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
DOCUMENT NUMBER: 53:83420
ORIGINAL REFERENCE NO.: 53:15069a-i,15070a
TITLE: Possible antiamebic agents. VI. Syntheses of some substituted dialkylaminotrimethylenebenzylamines and their dichloroacetates
AUTHOR(S): Sen, A. B.; Kulkarni, Yashwant D.
CORPORATE SOURCE: Univ. Lucknow
SOURCE: J. Indian Chem. Soc. (1958), 35, 205-9
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 35 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 48, 9371b. To 320 g. CH2:CHCN was added gradually 1 kg. 35% Me2NH at 40-5°; after 40 min. the mixture was saturated with NaOH yielding 82% Me2NCH2CH2CN, b. 169-72°; similarly was prepared Et2NCH2CH2CN, b20 86-8°, and (CH2)5NCH2CH2CN, b18 114-5°. To 225g. CH2:CHCN and a few drops of MeONa-MeOH was added 180 g. MeOH at 40-5°; on the following day acidification with AcOH gave 80.5% MeOCH2CH2CN, b. 162-4°; similarly were prepared: 78% EtOCH2CH2CN, b. 169-72°; 95% PrOCH2CH2CN, b24 85-9°; 85.5% BuOCH2CH2CN, b10 74-5; 95% EtSCH2CH2CN, b13 100°. These saturated with NH3 in ROH with ice cooling and hydrogenated over Raney Ni at 95-100° at 90-120 atmospheric H gave: 63.5% MeOCH2CH2CH2NH2, b734 117-19°; 50% EtOCH2CH2CH2NH2, b. 133-5°; 50% PrOCH2CH2NH2, b. 153-6°; 71% BuOCH2CH2CH2NH2, b21 74-6°. To 40 g. Na dispersed in MePh was added 36 g. EtSCH2CH2CN in 200 ml. dry EtOH, followed after dissolution of Na by 50 ml. EtOH and 200 ml. H2O; after acidification with HCl, concentration in vacuo, washing with Et2O, treatment with solid KOH, and extraction with Et2O there was obtained 28% EtSCH2CH2CH2NH2, b23 86-7°, nD20 1.4855, d20 0.9370. Saturation of 465 g. Me2NCH2CH2CN in 500 ml. MeOH with NH3 with cooling followed by hydrogenation over Raney Ni at 110 atmospheric H as above gave 68.8% Me2NCH2CH2CH2NH2 (I), b. 130-3°; similarly were obtained: 65% Et2NCH2CH2CH2NH2, b. 168-70°, and 50% (CH2)5NCH2CH2CH2NH2, b10 82-5°. To 10 g. I in 10 ml. H2O was added in 5 min. 13 g. MeCH:CHCOOMe:CH2 (mixed with corresponding methoxy ketones) in 10 ml. MeOH; after 8.5 hrs. at reflux the mixture was diluted and acidified with HCl, concentrated in vacuo, extracted with Et2O, treated with KOH, and extracted with Et2O yielding 64% 1-(3-dimethylaminopropyl)-2,5-dimethyl-4-piperidone, b2.5 96-7°, nD20 1.4726, d2020 0.9369 (di-HCl salt, m. 187-8°); a similar reaction in aqueous MeOH 6 hrs. at room temperature gave 87% above piperidone, while in MeOH an 8.5 hr. heating gave but 28% yield. All the piperidones described in this paper irritate the skin. Similarly, Et2NCH2CH2CH2NH2 gave in 8 hrs. at room temperature 77% 1-(3-diethylaminopropyl)-2,5-dimethyl-4-piperidone, b2 118-20°, 1.4678, 0.9146; (CH2)5NCH2CH2CH2NH2 similarly gave 57.5% 1-(3-piperidylaminopropyl)-2,5-dimethyl-4-piperidone, b1 126-7°, 1.4885, 0.9717. Similarly, MeOCH2CH2CH2NH2 in 4 hrs. in aqueous MeOH at room temperature gave 75% 1-(3-methoxypropyl)-2,5-dimethyl-4-piperidone, b3 110-11°, 1.4547, 0.9564 (HCl salt, m. 133-4°). This heated with NZH4.H2O in aqueous EtOH 5 hrs. at 70-5°, then freed of solvent, and heated with KOH fused in an Ag dish to 150-60° gave 53% 1-(3-methoxypropyl)-2,5-dimethylpiperidine, b2.5 60-2°, 1.4520, 0.8874 (HCl salt, m. 131.5-3°). Similarly were prepared: 68.7% 1-(3-ethoxypropyl)-2,5-dimethyl-4-piperidone, b2.5 116-18°, 1.4554, 0.9468 (HCl salt, oil); 41.5% 1-(3-ethoxypropyl-2,5-dimethylpiperidine, b2 58-60°, 1.4524, 0.8790 (HCl salt, oil); 67% 1-(3-propoxypropyl)-2,5-dimethyl-4-piperidone, b1.5 117-19°, 1.4545, 0.9357 (HCl salt, oil); 65% 1-(3-butoxypropyl)-2,5-dimethyl-4-piperidone, b2 127-9°, 1.4494, 0.9171 (HCl salt, oil); 68% 1-(3-ethylmercaptopropyl)-2,5-dimethyl-4-piperidone, b1.5 117-18°, 1.4915, 0.9896 (picrate, oil). Keeping 10 g. I, 16 g. 5-methyl-2,5-heptadien-4-one, 10 ml. H2O, and 20 ml. MeOH 6 hrs. at room temperature and 40 min. at 50-60° gave 72% 1-(3-dimethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5 104-6°, 1.4742, 0.9420. Similarly were prepared: 71.5% 1-(3-diethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5

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112-13°, 1.4736, 0.9309; 71% 1-(3-methoxypropyl)-2,5,6-trimethyl-4-piperidone, b1.5 97-8°, 1.4700, 0.9770; 70% 1-(3-ethoxypropyl)-2,5,6-trimethyl-4-piperidone, b2 110-11°, 1.4672, 0.9633; 73% 1-(3-propoxypropyl)-2,5,6-trimethyl-4-piperidone, b1.5 111-2°, 1.4645, 0.9510; 70% 1-(3-butoxypropyl)-2,5,6-trimethyl-4-piperidone, b2 119-20°, 1.4638, 0.9411. Similar reactions with propenyl 1-cyclohexenyl ketone similarly gave: 80% 1-(3-dimethylaminopropyl)-2-methyl-4-oxodecahydroquinoline, b2.5 138-40°, 1.4958, 0.9884; 81% 1-(3-diethylaminopropyl)-2-methyl-4-oxodecahydroquinoline, b2 146-7°, 1.4925, 0.9719; 64.5% 1-(3-piperidylpropyl)-2-methyl-4-oxodecahydroquinoline, b2 159-61°, 1.4963, 0.9854; 82.5% 1-(3-methoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5, 134-5°, 1.4951, 1.0219 (picrate, m. 133-5°); 81.7% 1-(3-ethoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5 140-2°, 1.4880, 1.0075 (this reduced as above with N2H4 gave 74% 1-(3-ethoxypropyl)-2-methyldecahydroquinoline, b3 119°, 1.4795, 0.9380 (HCl salt, m. 120-2°); 83% 1-(3-propoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2 142-3°, 1.4885, 0.9940; 74.5% 1-(3-butoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5, 151-2°, 1.4856, 0.9856.
ACCESSION NUMBER: 1957:85717 CAPLUS
DOCUMENT NUMBER: 51:85717
ORIGINAL REFERENCE NO.: 51:15520b-i,15521a-c
TITLE: Heterocyclic compounds. LII. Synthesis of 1-γ-alkoxypropyl-4-piperidones and 1-γ-dialkylaminoropyl-4-piperidones
AUTHOR(S): Nazarov, I. N.; Makin, S. M.
CORPORATE SOURCE: M. V. Lomonosov Inst. Fine Chem. Technol., Moscow
SOURCE: Zhurnal Obshchei Khimii (1957), 27, 499-509
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 36 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB See C.A. 49, 3034c.
ACCESSION NUMBER: 1955:23844 CAPLUS
DOCUMENT NUMBER: 49:23844
ORIGINAL REFERENCE NO.: 49:4616d-e
TITLE: Cyanoethylation of cyclic and heterocyclic alcohols and amines. Hydrogenation and alcoholysis of products of cyanoethylation
AUTHOR(S): Nazarov, I. N.; Shvekhgeimer, G. A.
SOURCE: Zhurnal Obshchei Khimii (1954), 24, 165-70
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: English

L3 ANSWER 37 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB Unavailable
ACCESSION NUMBER: 1955:23843 CAPLUS
DOCUMENT NUMBER: 49:23843
ORIGINAL REFERENCE NO.: 49:4616c-d
TITLE: The conjugate addition of tert-butylmagnesium chloride to o-hydroxy diaryl ketones
AUTHOR(S): Fang, Fabian T.
CORPORATE SOURCE: Univ. of Illinois, Urbana
SOURCE: (1955) 64 pp.; microfilm, $1.00; paper enlargement, $6.40 Avail.: Univ. Microfilms (Ann Arbor, Mich.), Order No. 10470
From: Dissertation Abstr. 15, 38
DOCUMENT TYPE: Dissertation
LANGUAGE: Unavailable

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AB Addition of 105 g. CH2:CHCN to 193 g. cyclohexanol and 14 g. 40% KOH with cooling below 30°, followed by stirring 6 h. at room temperature and standing overnight, gave, after neutralization with dilute HCl and filtration of KCl, 208 g. C6H11OCH2CH2CN, b20 130-2°, nD20 1.4586, d20 0.9674; this (35 g.) hydrogenated in MeOH saturated with NH3 over Raney Ni at 95-105° and 145 atmospheric H to 34.5 g. C6H11OCH2CH2CH2NH2, b4.5 72-4°, nD20 1.4646, d20 0.9281 (phenylcarbamide, m. 101.5-2.5°). Similarly 142 g. 2-methylcyclohexanol gave with 70 g. CH2:CHCN and 10 g. 40% KOH, 137.5 g. 2-methylcyclohexyl 2-cyanoethyl ether, b18 134-7°, nD20 1.4564, d20 0.9502, which hydrogenated as above to 2-methylcyclohexyl 3-aminopropyl ether, b3.5 73-4.5°, nD20 1.4603, d20 0.9115 (phenylcarbamide, m. 96-8°). Similarly 114 g. 3-methylcyclohexanol and 53 g. CH2:CHCN gave 109.5 g. 3-methylcyclohexyl 2-cyanoethyl ether, b16 133-6°, nD20 1.4529, d20 0.9458, which hydrogenated to 3-methylcyclohexyl 3-aminopropyl ether, b4 76-8°, nD20 1.4599, d20 0.9118. To 45 g. 1,2,5-trimethyl-4-piperidinol and 3 g. 40% KOH was added 17 g. CH2:CHCN (temperature rise to 35° observed) and the mixture stirred at room temperature 4 h., then allowed to stand overnight; there was obtained 42 g. 1,2,5-trimethyl-4-piperidyl 2-cyanoethyl ether (I), b4 117°, nD20 1.4635, d20 0.9661 (picrate, m. 111-13°), which hydrogenated to 1,2,5-trimethyl-4-piperidyl 3-aminopropyl ether, b8 116-18°, nD20 1.4680, d20 0.9315 (picrate, m. 147-9°). I (20 g.), 50 mL. EtOH, and 30 g. concentrated H2SO4 stirred 18 h. at 90° gave after dilution, neutralization, and extraction with Et2O 20.2 g. 1,2,5-trimethyl-4-piperidyl 2-carbethoxyethyl ether, b4 109-10°, nD20 1.4504, d20 0.9807 (HCl salt, m. 93.5-5°); similar run in MeOH at 70° failed to react in 8 h. To 122 g. MeNH2 in 700 mL. MeOH was added over 2 h. 208 g. CH2:CHCN at below 30°; after stirring 10 h. at room temperature the mixture gave 255.5 g. MeNHCH2CH2CN, b25 86°, nD20 1.4320, and 54 g. MeN(CH2CH2CN)2, b6 162-4°, nD20 1.4606. Heating 21.5 g. MeNHCH2CH2CN with 16 g. CH2:CHCN 16 h. at 90° gave 31.2 g. MeN(CH2CH2CN)2, b5 159.5-60°, nD20 1.4612. Hydrogenation of the latter in MeOH saturated with NH3 over Raney Ni at 90-100° and 100 atmospheric H gave MeN(CH2CH2CH2NH2)2, b4 81-3°, nD20 1.4753, d20 0.9023, along with some 1-methyl-1,5-diazacyclooctane obtained in the low b. fraction [HCl salt, m. 189.5-90° (from EtOH)]. To 21 g. MeNHCH2CH2CN was added with cooling 21.5 g. CH2:CHCO2Me and the mixture gave after 5 days at room temperature 39.3 g. MeN(CH2CH2CN)CH2CH2CO2Me, b6 133°, b4.5 114°, nD20 1.4507, d20 1.0338 [picrate, m. 108-9° (from EtOH)]. Hydrogenation of this in MeOH saturated with NH3 over Raney Ni at 90-100° and 100 atmospheric H gave a viscous mass which polymerized to a rubbery mass. To 270 g. Me2NH and 400 mL. MeOH was added with cooling 318 g. CH2:CHCN at below 30°; after 1 h. at 40-55° and standing overnight the mixture yielded 566.5 g. Me2NCH2CH2CN, b18 68°, b10 60-1°, nD20 1.4282, which hydrogenated to Me2NCH2CH2CH2NH2, b758 131-4°, nD20 1.4398 (HCl salt, m. 183-4°). Heating 127 g. 2,5-dimethyl-4-piperidone with 53 g. CH2:CHCN 3 h. at 95-7° and allowing the mixture to stand overnight gave 104 g. starting material and 31.2 g. 1-(2-cyanoethyl)-2,5-dimethyl-4-piperidone, b5.5 143-5°, nD20 1.4841, d20 1.0345 [HCl salt, m. 166.5-7° (from Me2CO); picrate, m. 136-7° (from Me2CO)]. This hydrogenated to 1-(3-aminopropyl)-2,5-dimethyl-4-aminopiperidine, b3.5 108-10°, nD20 1.4917, d20 0.9475 (picrate, m. 227.5-8.5°), under conditions stated above.
ACCESSION NUMBER: 1955:15753 CAPLUS
DOCUMENT NUMBER: 49:15753

L3 ANSWER 38 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
ORIGINAL REFERENCE NO.: 49:3034c-i,3035a
TITLE: Cyanoethylation of cyclic and heterocyclic alcohols and amines. Hydrogenation and alcoholysis of products of cyanoethylation
AUTHOR(S): Nazarov, I. N.; Shvekhgeimer, G. A.
CORPORATE SOURCE: Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow
SOURCE: Zhurnal Obshchei Khimii (1954), 24, 163-9
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 39 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 41, 1609h; 42, 3722g. Dissertation at the University (1946) with complete exptl. details and bibliography of 169 references. A laboratory preparation of CH2:CHCN (I) was developed as follows. To a hot saturated solution of 100 g. SnCl2 was added 30 g. Zn dust with stirring and, after completion of reaction, the mixture was allowed to stand 2 hrs., decanted, washed with 10% AcOH, let stand overnight with 60 ml. 80-90% AcOH, filtered, washed with H2O until neutral, and washed with EtOH and Et2O, giving 30-35 g. Sn dust. All traces of Zn must be removed for good results with this catalyst. Heating 50 g. HOCH2CH2CN with 5 g. of the above Sn dust in a distillation apparatus with chilled receiver so that vapor temperature is below 110° yields a 2-layer distillate; the upper layer after drying with CaCl2 yields up to 90% I. If con. ethylene oxide is used in the preparation of the cyanohydrin, the product may be contaminated with MeCH:CHCN, H2O, and NH3; it is purified by 5-10 min. treatment with P2O5 and distillation (b758 78°). Refluxing the cyanohydrin with silica gel, activated C, MgSO4, Fe oxides, pieces of sheet Fe, Al foil, and Al2O3 gave but 0-30% yields of I. Passage of the cyanohydrin over Al2O3 at 200-20° gave but 18-20% I. To 950 ml. aqueous NH4OH (saturated in the cold) was added 95 g. I dropwise with cooling over 2 hrs. so that the mixture remained homogeneous; after 30 min. at room temperature, distillation gave 30% H2NCH2CH2CN, b14 77-8°, b23 89°, nD20 1.4390, d20 0.9584, which polymerized in several days in a sealed ampul even in darkness. Distillation of the higher-boiling residue gave 47% HN(CH2CH2CN)2, b14 177-9°, b22 209-11°, nD20 1.4630, d20 1.0196; HCl salt, m. 147-8° (from MeOH); N-Bz derivative, m. 112° (from MeOH). The free amine generated by addition of 50% aqueous Me2NH to solid NaOH was fed into 106 g. I with ice cooling over 6-8 hrs., and the mixture distilled after 2 hrs. at room temperature yielding 80-1% Me2NCH2CH2CN, b750 171°, nD20 1.4283, d20 0.8705; picrate, m. 151°; HCl salt, m. 199° (from MeOH). A mixture of 40 g. Et2NH and 26.5 g. I gave a slight heat evolution after 5-10 min.; refluxed on a steam bath 2 hrs. (yellow color) and distilled, it yielded 89-95% Et2NCH2CH2CN, b20 86-9°. If the heating is done in sealed tubes 6-8 hrs. no yellow color is formed and the yield is nearly 100%; the pure product b2 65°, b9 76°, b20 87°, b45 112°, b755 197.3° (corr.), d20 0.8761, nD20 1.4380; HCl salt, m. 120°; picrate, m. 85°. This (3.1 g.) refluxed 4 hrs. with 4. g. 25% NaOH and evaporated gave the amorphous Na salt of the corresponding acid; refluxing 6.3 g. of the nitrile with 11 g. concentrated HCl, cooling, filtering, and evaporating repeatedly in vacuo gave an amorphous mass, which was freed in aqueous solution of Cl ion by Ag2CO3, the Ag ion removed with H2S, and the filtrate evaporated, yielding 60% Et2NCH2CH2CO2H, m. 70-5°. The best reaction conditions for piperidine and I are as follows: Piperidine (17 g.) and 11.1 g. I mixed with cooling in an ampul (cooled until the heat evolution stopped in 15-20 min.) and heated 4 hrs. on a steam bath, then let stand overnight, gave 96-7% (CH2)5NCH2CH2CN, b18 114-15°; some 22% is formed by refluxing 5 g. piperidine with 5 g. HOCH2CH2CN 3 hrs. at 120-50°; if Sn dust is added the yield is 52.5%. An extensive study showed that the reaction of I with PhNHEt is best carried out by heating in an ampul 100 hrs. on steam bath in the presence of 3% Ac2O and a little hydroquinone, when 65-70% PhEtNCH2CH2CN, b8 158°, b11 164-5°, nD20 1.5503, d20 1.0260, is obtained; HCl salt, hygroscopic solid; picrate, oil; the free base couples with

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diazotized sulfanilic acid even in acid medium and the coupling product, isolated as the Na salt, is a green solid, giving a brown color in acid soln. Coupling with diazotized p-O2NC6H4NH2 gave a brown product, C17H17O2N5, while tetrazotized benzidine reacts only slowly in acidified soln., yielding a red-violet soln. which turns yellow in neutral or basic soln.; the free azo deriv. is sol. in org. solvents. Hydrolysis of PhEtNCH2CH2CN is very slow with H2O at 100° in a sealed tube; concd. HCl at room temp. acts slowly and incompletely even in 48 hrs., while heating at 110-20° leads to loss of PhNHEt; heating with 30-40% H2SO4 gives an impure product. Alk. hydrolysis gives low yields of the corresponding acid. Refluxing 14 g. PhEtNCH2CH2CN and 20 g. KOH in 20 ml. H2O and 70 ml. EtOH 15 hrs., acidifying with HCl, and repeatedly extg. with iso-BuOH, adding Et2O to the ext. gave 33.1% PhEtNCH2CO2H.HCl, a high-melting solid, giving a brown color with FeCl3. This couples even in acid soln. with diazotized sulfanilic acid, yielding a red azo deriv.; p-O2NC6H4N2Cl also couples in acid medium, giving a red azo deriv. PhEtNCH2CH2CN (4.5 g.) added slowly to 15 ml. concd. H2SO4, and the mixt. let stand 40 hrs., then dild. with H2O (50 ml.), neutralized with concd. NH4OH, and let stand overnight giving a ppt. of PhEtNCH2CH2CONH2, 68.5-76.5%, m. 55-8° (crude), m. 67° (from MeOH). I (35 g.) added to 20 g. dry (CH2NH2)2 dropwise with cooling at 15-20° over 2 hrs. the mixt. shaken 2 hrs. at room temp. and let stand overnight in a stoppered flask gave 39.8% H2NCH2CH2NHCH2CH2CN, b1.5 101°, nD20 1.4727, d20 0.9912 (with Me2nI at room temp. only the primary amino group reacts, while at 100° all active H can be detd.) (the picrate and styphnate are oils, while HCl salt is a viscous mass), and 59.8% (CH2NHCH2CH2CN)2, b1.5 174°, b3.5 191°, nD20 1.4793, d20 1.0256 [picrate and styphnate, oils; HCl salt, m. 184-7° (decompn.)]. The structure of the latter appears confirmed by the improbability of reaction of I with a cyanoethylated group, and further by the reaction with MeZnI which indicates 1.94 active H atoms/mole at 100° and 0.5 at room temp. Me2NCH2CH2CN treated with MeI in C6H6 with cooling gave the methiodide, m. 153° (from MeOH); EtI at room temp. yielded the ethiodide, m. 128.5° (from MeOH); EtBr at 60° yielded the ethobromide, m. 157° (from Et2O-MeOH); PrBr and CH2:CHCH2Cl at 80° yielded the corresponding quaternary salts, m. 189° (from Et2O-MeOH), and 185-7° (from MeOH), resp. Et2NCH2CH2CN with MeI at room temp. gave the methiodide, m. 152° (from MeOH), while EtI at 60° gave the ethiodide, m. 168° (from MeOH). (CH2)5NCH2CH2CN with MeI at 100° gave the methiodide, m. 152° (from MeOH), while EtI reacted slowly at 100° yielding the ethiodide, m. 160-1° (from MeOH). Reduction of H2NCH2CH2CN with BuOH-Na gave variable yields when com. Na was used, because of traces of K (Dzirkal, C.A. 36, 2255.6); a 2% K-Na alloy gave high yields comparable to those obtained with pure Na. In the best procedure 30 g. of this alloy was rapidly treated with 14 g. H2NCH2CH2CN in 450 ml. BuOH, and despite vigorous reaction the mixt. was immediately heated in an oil bath at 140-50°, cooled after 35-40 min., dild. with 130-50 ml. cold H2O, steam-distd. 4-6 hrs. into the calcd. amt. of aq. HCl, and the distillate evapd., yielding 81% CH2(CH2NH2)2.2HCl, m. 242° (from EtOH). Similar reduction of Me2NCH2CH2CN gave 52-6% Me2NCH2CH2 CH2NH2, b128-30 70-80° (crude), b20 44-5°, b748 133°, nD20 1.4415, d20 0.8272; di-HCl salt, m. 184° (from MeOH); picrate, C17H20N8O14, m. 211° (from H2O). The higher-boiling material yielded a little 3,3'-bis(dimethylamino)dipropylamine, b20 128-31°, nD20 1.4531 (HCl salt, hygroscopic solid; tripicrate, m. 200°; chloroplatinate, 2C10H25N3.3H2PtCl6, sol. in H2O, insol. in aq. EtOH). Reduction of Et2NCH2CH2CN with NaBuOH gave 38-63% diamine; a 2% K-Na alloy gave good

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consistent 60-70% yields; pure Et2NCH2CH2CH2NH2, b12 61-2°, b70 85-7°, b80 99-100°, b755 168-70°, nD20 1.4435, gave 2 active H with MeZnI at room temp. and at 100°; picrate, m. 190.5° (from MeOH); Bz deriv., oil. Refluxing this amine with an equimolar amt. of oleic acid 2 hrs., adding a little amine, heating another hr., concg., and evapg. with C6H6 gave a product that formed extremely stable org.-aq. emulsions. The higher-boiling fractions from the above reduction gave a little bis(diethylamino) dipropylamine, b12 148-50° (picrate, m. 152°), also obtained if the reduction is run with pure Na. Reduction of (CH2)5NCH2CH2CN with 2% K-Na in BuOH gave 57% 1-(3-aminopropyl)piperidine, b4 65-6°, b9 79-81°, nD20 1.4729. COCl2 with ROH gave the ClCO2R: R =Et, b752 92-4°; Pr, b742 114-16°, nD20 1.4036; iso-Pr, b745 101-2°, nD20 1.3996, d20 1.0777; Bu, b16 40-7°, b756 138°, nD20 1.4128, d20 1.0513. COCl2 with ROH in MePh in the presence of 5-8% quinoline gave the following ClCO2R: iso-Bu, b750 123-7°; iso-Am, b754 150-1°, nD20 1.4176, d20 1.0490; C8H17, b5 86.5°, b10 96-7°, b15 107°, nD20 1.4330, d20 0.9841; cyclohexyl, b25 80-5°, nD25 1.4628; 1-menthyl, b5 96°, b11 108-9°, nD20 1.4712; PhCH2, b7 85-7°; with an equimolar amt. of quinoline were obtained: sec-Bu, 72%, b23 30-1°, b748 121-4°, nD20 1.4490; 1-methyl-2-cyclohexyl, b30 101.5°, nD20 1.4560; Ph, b7 64°, nD20 1.5162. The diamines (0.025 mole) in Et2O were treated with 0.025 mole powd. potash, then 1.5-2 ml. H2O, and RO2CCl in Et2O was added with cooling; the usual treatment gave the desired urethan derivs.: Me2NCH2CH2CH2NHCO2Et, 55.8%, b16 137-7°, nD20 1.4480, d20 0.9653; 1-menthyl ester, 51.8%, b1 164.5°, nD20 1.4706, d20 0.9557, m. 45°; Et2NCH2CH2CH2NHCO2Et, 66.7%, b7 130°, nD20 1.4503; iso-Pr ester, 53.2%, b1.5 122-3°, nD30 1.4452, nD20 1.4493, d20 0.9367; sec-Bu ester, 42.5%, b5 132°, nD20 1.4513, d20 0.9334; C8H17 ester, 63.3%, b2 181.5-2°, nD30 1.4528, nD20 1.4577, d20 0.9168; cyclohexyl ester, 46.5%, b1.5 165-7°, nD30 1.4725, nD20 1.4752, d20 0.9765; 2-methylcyclohexyl ester, 81.5%, b2 177°, nD30 1.4693, nD20 1.4723, d20 0.9679; 1-menthyl ester, 88.2%, b3 173°, nD20 1.4719, d20 0.9482, m. 31°; Ph ester, 33.6%, b3 196-201°, nD20 1.4770; PhCH2 ester, 24%, b3 132-5°, nD20 1.5030. C5H5NCH2CH2CH2NHCO2Et, 78.3%, b9 150-3°, nD20 1.4742, d20 1.0070; Pr ester, 70.4%, b18 187-8°, nD20 1.4735, d20 0.9935; iso-Pr ester, 62.8%, b8 155-8°, nD20 1.4706, d20 0.9878; Bu ester, 62.8%, b3 146°, b5 167-8°, nD20 1.4730, d20 0.9788; iso-Bu ester, 53.5%, b2 136.5-7°, nD20 1.4710, d20 0.9813; iso-Am ester, 66.2%, b2 159.5°, nD20 1.4712, d20 0.9749; C8H17 ester, 63.7%, b9 212-13°, nD20 1.4720, d20 0.9550.
ACCESSION NUMBER: 1953:58498 CAPLUS
DOCUMENT NUMBER: 47:58498
ORIGINAL REFERENCE NO.: 47:9905i,9906a-i,9907a-i,9908a-b
TITLE: Acrylonitrile as a starting material for synthesis of amino nitriles and polyamines
AUTHOR(S): Kost, A. N.
SOURCE: Uchenye Zapiski Moskov. Gosudarst. Univ. im. M. V. Lomonosova (1950), (No. 131), 39-97
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB Compds. useful as wetting agents, detergents, emulsifying agents, germicides, and fungicides are prepared as follows. (HOCH2CH2)2NH 250 is added slowly to CH2:CHCN (I) 126, and the solution heated 2.4 h. on a steam bath and vacuum-distilled at 100° leaves (HOCH2CH2)2NCH2CH2CN (II) 356 g. II 350 hydrogenated at 2000 lb./sq. in. and 115° in the presence of Raney Ni 10 g. and NH3 4.4 mol gives 76% (HOCH2CH2)2N(CH2)3NH2 (III), b8.6 175-87°. III 33 and stearic acid 56.8 in PhMe 50 refluxed 9 h. at 155-61° give C17H35CONH(CH2)3N(CH2CH2OH)2 (IV) 88 g. HCl 22.4 cc. added to IV 105 g. in EtOH 225 cc., and the solution heated to 62°, treated with ethylene oxide (V) 16 g., and heated 4 h. at 100° give a compound useful as an assistant for stripping vat dyes from cellulosic textiles and as a rewetting agent. HCl 90.5 cc. added to C7H15CONH(CH2)3NMe2 250 in EtOH 450, and the solution heated 3 h. at 50° with V 53 gives C7H15CONH(CH2)3N(Cl)(Me2)CH2CH2OH 340 g. I 170 and 25% aqueous Me2NH 545 give Me2NCH2CH2CN 218 g., which is hydrogenated to Me2N(CH2)3NH2 (VI), b. 134°. Me(CH2)12COCl 38 added dropwise to VI 15.5 in C6H6 160 g. and the solution stirred 1 h. gives C13H27CONH(CH2)3NMe2 (VII), b1-2 215°. V 40 added to VII 266 in EtOH 450 g. and HCl 90.5 cc. and the solution heated 3 h. at 80° gives C13H27CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C13H27CONHCH2CH2NMe2 7.4 and ClCH2CH2OH (VIII) 2 g. in EtOH heated 3 h. at 130-40° give C13H27CONHCH2CH2N(Cl)(CH2CH2OH)Me2. Wood rosin acid 221 and VI 100 g. heated at 200-15° give N-(3-dimethylaminopropyl)abietamide (IX). V 12 passed into IX 93 in alc. 93 g. and HCl 20 cc. and the mixture let stand overnight at 46° gives (3-abietoylaminopropyl)dimethyl(2-hydroxyethyl)ammonium chloride. C17H35CONH(CH2)3NMe2 (X) 206 in EtOH 300 g. adjusted to pH 3.9 with HCl, heated to 40-50°, and treated with V gives C17H35CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C11H23CONH(CH2)3NMe2 468 and VIII 133 heated 2 h. at 125° give C11H23CONH(CH2)3N(Cl)(CH2CH2OH)Me2. X 117 and ClCH2CH2(OH)CH2OH 35 g. stirred 1.5 h. at 125° give C17H35CONH(CH2)3N(Cl)(CH2CH(OH)CH2OH]Me2.

ACCESSION NUMBER: 1952:67119 CAPLUS
DOCUMENT NUMBER: 46:67119
ORIGINAL REFERENCE NO.: 46:11227i,11228a-e
TITLE: Aliphatic amido propyl quaternary ammonium salts
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2589674		19520318	US	

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AB Quaternary ammonium compds. of the type RCONH(CH2)3N(X)R1R2R3, in which R1 is an alkyl group of at least 7 C atoms, R2 an alkyl group of lower mol. weight, R3 an alkyl, aralkyl, or aliphatic olefin, and X an anion, are prepared by reaction of an appropriate tertiary amine with an alkyl halide, dialkyl sulfate, etc. The new compds. are soluble in H2O, practically odorless, relatively nontoxic to man, and are useful as antiseptics, wetting agents, and emulsifiers. An aqueous solution of Me2NH (25%) 545 was treated with CH2:CHCN 170 parts at a temperature below 20°, left standing for 1 hr., mixed with 350 cc. aqueous NaOH (10%), the aqueous layer extracted with Et2O, and the Et2O removed to yield 218 parts of 2-Me2NC2H4CN (I), b22 73-4°. Hydrogenation of I at 100° and 90 atmospheric pressure in the presence of anhydrous NH3 with Raney Ni as catalyst gave N,N-dimethylpropylenediamine (II), b. 134°. A solution of II 15.5 in C6H6 160 was treated with C13H27COCl 38 parts, stirred for 1 hr., washed with aqueous NaOH (10%) and H2O, and distilled in vacuo to give (3-myristoylaminopropyl)dimethylamine (III), b1-2 208-15°. A solution of III 6.2 and PhCH2Cl 3.4 in C6H6 30 parts was refluxed for 4 hrs. and yielded after removal of the solvent (3-myristoylaminopropyl)dimethylbenzylammonium chloride, m. 54°; this compound forms a clear 25% solution in H2O and is a germicide effective against Staphylococcus aureus in a dilution of 1:25,000 at 37° in a 5-min. test and has a PhOH coefficient of 277-333; it is also a wetting agent for cotton fabrics. Using the same procedure, (3-lauroylaminopropyl)dimethylbenzylammonium chloride is obtained, which also is a good germicide.

ACCESSION NUMBER: 1949:23628 CAPLUS
DOCUMENT NUMBER: 43:23628
ORIGINAL REFERENCE NO.: 43:4430h-i,4431a-b
TITLE: Quaternary ammonium compounds
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459062		19490111	US	

L3 ANSWER 42 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB (Stearoylaminopropyl)dimethylbenzylammonium chloride was synthesized in 5 steps: (1) 25% aqueous Me2NH 545 g. was added to CH2:CHCN 170 g. below 20°, poured after 1 hr. into 350 cc. 10% aqueous NaOH, and the oily layer plus the ether extract dried and distilled to yield 218 g. of β-(dimethylamino)propionitrile, b22 73-4°. (2) Me2N(CH2)2CN 207 hydrogenated over Raney Ni at 100° and 90 atmospheric in the presence of NH3 72.4 yielded 3-(dimethylamino)propylamine, b760 134°, 204.5 g. (3) C17H35COCl 49 was added dropwise to Me2N(CH2)3NH2 15.5 in C6H6 160 g. and the solution was washed after 1 hr. with 10% aqueous NaOH and H2O and distilled, giving a solid N,N-dimethyl-3-(stearoylamino)propylamine, b1-2 208-15°. (4) C17H35CONH(CH2)3NMe2 0.4 mol. was treated with C2H4O in the presence of 0.93 g. NaOH in tert-BuOH at 65°, and the NaOH neutralized with 1.9 cc. 38% HCl. (5) The product of step (4) was quaternized by reaction with 51 g. PhCH2Cl at 75° 2 hrs.; after filtering and evaporating off the solvent the quaternary amine salt was a crystalline solid at room temperature, soluble in aqueous Na2CO3 or H2O. Similar products are made using capryl, lauroyl or palmitoyl chloride in step (3) or 1-C10H7CH2Cl in step (5).

ACCESSION NUMBER: 1949:13222 CAPLUS
DOCUMENT NUMBER: 43:13222
ORIGINAL REFERENCE NO.: 43:2630i,2631a-c
TITLE: Aliphatic amide-substituted propyl quaternary ammonium compounds
INVENTOR(S): Moss, Philip H.; Cook, Elmer W.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459088		19490111	US	

L3 ANSWER 43 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dissertation summary. CH2:CHCN (I) was condensed with amines to β-amino nitriles which were hydrolyzed to the acids for phytohormone studies; the nitriles were also condensed with RX to quaternary N compds. for bactericidal studies; the aromatic derivs. were coupled with diazonium compds. to new dyes. Reduction methods were studied to give diamine derivs. Aliphatic amines add to I in quant. yield with exothermal reaction; PhNHEt required the following method for 70% yield: the reagents are heated 100 hrs. in a sealed tube to 100° in the presence of Ac2O and a little hydroquinone. Hydrolyses were done at reflux in dilute alc. KOH. Reductions were done with Na-K alloy in BuOH. The diamine urethans were made using esters of ClCO2H in moist Et2O in the presence of K2CO3. The exptl. work was done in 1938-41, thus anticipating Whitmore, et al. (C.A. 38, 3617.3). The compds. made were: H2NCH2CH2CN (34-6%), b23 89°, nD20 1.4390, d420 0.9584; HN(CH2CH2CN)2 (57-60%), b14 177-9°, nD20 1.4610, d420 1.0196 (Ac derivative, m. 146°; Bz derivative, m. 112°; picrate, oil); Me2NCH2CH2CN (80-1%), b750 171°, nD20 1.428, d420 0.8703 (picrate, m. 151°; HCl salt, m. 199°; methiodide, m. 153°; ethiodide, m. 128.5°; ethobromide, m. 157°; propobromide, m. 189°; allochloride, m. 185-7°); Et2NCH2CH2CN (96-8%), b2 65°, b9 76°, b20 87°, b755 197.3°, nD20 1.4380, d420 0.8761 (picrate, m. 85°; HCl salt, m. 120°; methiodide, m. 152°; ethiodide, m. 168°); 2-(1-piperidyl)-1-cyanoethane (96-7%), b18 114-15° (methiodide, m. 152°; ethiodide, m. 160-1°); N-ethyl-N-(2-cyanoethyl)aniline (70%), b8 158°, nD20 1.5503, d420 1.0260 (picrate and styphnate, oils; amide, prepared after hydrolysis, m. 67°); H2NCH2CH2NHCH2CH2CN (39.8%), b1.5 101°, nD20 1.4727, d420 0.9912; (CH2NHCH2CH2CN)2 (59%), b1.5 174°, nD20 1.4792, d420 1.0256 (HCl salt, m. 184-7° (decomposition)); H2NCH2CH2CH2NH2 (81%, isolated as the HCl salt, m. 242°); Me2NCH2CH2CH2NH2 (52-6%), b748 133°, nD20 1.4415, d420 0.8272 (HCl salt, m. 184°; picrate, m. 211°); HN(CH2CH2CH2NMe2)2, b20 128-31°, nD20 1.4531 (picrate, m. 200°); Et2NCH2CH2CH2NH2 (60-70%), b80 99-100°, nD20 1.4425 (picrate, m. 190.5°; Bz derivative, oil); HN(CH2CH2CH2NEt2)2, b12 148-50° (picrate, m. 152°); 1(3-aminopropyl)piperidine (57%), b9 79-81°, nD20 1.4729. R2NCH2CH2CH2NHCO2R' (R and R' given): Me, Et, b6 137-8°, nD20 1.4480, d420 0.9653; Me, l-menthyl, b11 164.5°, nD20 1.4706, d420 0.9557. Et2NCH2CH2CH2NHCO2R (R given): Et, b7 130°, nD20 1.4503; iso-Pr, b1.5 122-3°, nD20 1.4493, d420 0.9367; sec-Bu, b5 132°, nD20 1.4513, d420 0.9334; C8H17, b2 181.5-2°, nD20 1.4577, d420 0.9168; cyclohexyl, b1.5 165-7°, nD20 1.4752, d420 0.9765; 1-methylcyclohexyl, b2 177°, nD20 1.4723, d420 0.9679; l-menthyl, b3 173°, m. 31°, nD20 1.4719, d420 0.9482; benzyl, b3 132-5°, nD20 1.5030; Ph, b3 196-201°, nD20 1.4770. (CH2)5NCH2CH2CH2NHCO2R: Et, b9 150-3°, nD20 1.4742, d520 1.0070; Pr, b18 187-8°, nD20 1.4735, d420 0.9935; iso-Pr, b8 155-8°, nD20 1.4706, d420 0.9878; Bu, b3 146°, nD20 1.4730, d420 0.9788; iso-Bu, b2 136.5-7°, nD20 1.4710, d420 0.9813; iso-Am, b2 159.5°, nD20 1.4712, d420 0.9749; C8H17, b9 212-13°, nD20 1.4720, d420 0.9550.

ACCESSION NUMBER: 1948:17366 CAPLUS
DOCUMENT NUMBER: 42:17366
ORIGINAL REFERENCE NO.: 42:3722g-i,3723a-e
TITLE: Acrylonitrile as source material for the synthesis of amino nitriles and polyamines
AUTHOR(S): Kost, A. N.
SOURCE: Vestnik Moskovskogo Universiteta (1947), No. 2, 141-6
CODEN: VMUNAE; ISSN: 0372-6320

L3 ANSWER 43 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L3 ANSWER 44 OF 44 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 42, 1566i. Tech. Me2NH or Me2NH sulfate in 50% aqueous solution was poured in a thin stream onto solid NaOH, and the Me2NH so generated was passed over soda lime, then through a series of absorption flasks containing 106 g. dry CH2:CHCN, cooled with snow; absorption was complete in 6-8 hrs., and after an addnl. 20 min. passage of the amine, the solns. were allowed to stand 2 hrs. and were distilled to give 80-1% β-dimethylaminopropionitrile, b745 168-70°; on redistn., it b750 171°, d420 0.8705, nD20 1.4283; picrate, m. 151° (from MeOH); HCl salt, m. 199° (from MeOH); methiodide, m. 153° (from MeOH); ethiodide, m. 128.5° (from MeOH); ethobromide, m. 157° (from MeOH-Et2O); propobromide, m. 189° (from MeOH Et2O); allochloride, m. 185-7° (from MeOH). The nitrile (20 g.) in 450 cc. dry BuOH was rapidly added to 30 g. Na containing 2% K and the mixture was immediately heated so that the solution was complete in 20-5 min. (use of pure Na triples the reaction time). On cooling, 100 g. H2O was added and the BuOH distilled with steam, which also removed the diamine; a calculated amount of 2 N HCl was used in the receiver. The distillate was slowly concentrated with a stream of steam and auxiliary heating to remove the BuOH and most of the H2O; the residue, treated with solid NaOH, extracted with Et2O, and the extract dried over NaOH, gave 52-6% 1-dimethylamino-3-aminopropane (I), b128-30 70-80°; redistn. gave the pure diamine, b20 44-5°, b748 133°, d420 0.8272, nD20 1.4415; HCl salt, m. 184° (from MeOH); picrate, m. 211° (from EtOH). If the reduction is done with pure Na there is formed 1-2.2 g. higher-boiling fraction, from which can be isolated 0.4 g. bis(3-dimethyl-aminopropyl)amine, b20 128-31°, nD20 1.4531; HCl salt, hygroscopic flakes (from Et2O-HCl); picrate, m. 200° (from water); chloroplatinate, orange cubes. I (3.1 g.) in 30 cc. Et2O and 4.1 g. K2CO3 were treated with 1.5 cc. water, followed by 3.3 g. ClCO2Et in 30 cc. Et2O added slowly with cooling; after stirring 8 hrs. and standing overnight, distillation of the organic solution gave 55.8% Et 3-dimethylaminopropylcarbamate, b16 137-8° d420 0.9653, nD20 1.4480. l-Menthyl chloroformate gave 51.8% l-menthyl analog, b1 164.5°, d420 0.9557, nD20 1.4706; on standing 5 yrs. this solidified and m. 45° (from dilute MeOH).
ACCESSION NUMBER: 1948:11880 CAPLUS
DOCUMENT NUMBER: 42:11880
ORIGINAL REFERENCE NO.: 42:2578d-i
TITLE: Syntheses with acrylonitrile. VI. Preparation of 1-dimethylamino-3-aminopropane
AUTHOR(S): Terent'ev, A. P.; Kost, A. N.
CORPORATE SOURCE: Moscow State Univ., Moscow, USSR
SOURCE: Zhurnal Obshchei Khimii (1947), 17, 1632-6
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Russian